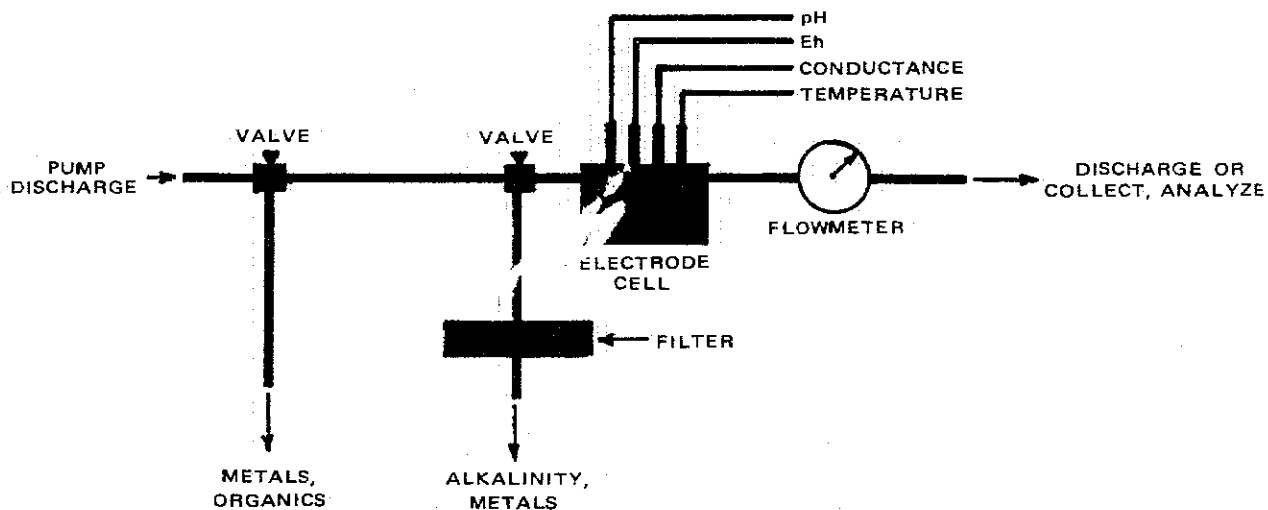




QUALITY ASSURANCE INTERIM GUIDELINES

FOR WATER QUALITY SAMPLING AND ANALYSIS:
GROUND WATER MANAGEMENT AREAS PROGRAM



WATER QUALITY INVESTIGATIONS SECTION

DECEMBER 1986

State of
Washington

Booth Gardner
Governor

Department
of Ecology

Andrea Beatty Riniker
Director

QUALITY ASSURANCE INTERIM GUIDELINES
FOR WATER QUALITY SAMPLING ANALYSIS:
GROUND WATER MANAGEMENT AREAS

December 1986

Primary Author
Barbara Carey

Washington State Department of Ecology
Water Quality Investigations Section
Olympia, WA. 98504-6811

TABLE OF CONTENTS

<u>Section Number</u>	<u>Title</u>	<u>Page Number</u>
	List of Figures	iii
	List of Tables	iv
1	INTRODUCTION	1
2	PROJECT DESCRIPTION	4
3	PROJECT ORGANIZATION AND RESPONSIBILITIES	5
4	OBJECTIVES FOR MEASUREMENT DATA	10
5	SAMPLING AND FIELD MEASUREMENT	12
	5.a. Sample Container Preparation and Preservatives	12
	5.b. Field Notes	12
	5.c. Calibration Procedures and Frequency for Field Instruments and Preventive Maintenance	18
	5.d. Design and Construction Guidelines for Monitoring Wells	18
	5.e. Static Water Level Measurement	18
	5.f. Well Purging Prior to Sample Collection	25
	5.g. Sampling Mechanics	29
	5.h. Field Analyses	38
	5.i. Field Quality Control Checks (blanks, duplicates, split samples, and standards)	39
	5.j. Sample Storage, Packaging, and Transport	43
6	ANALYTICAL SYSTEM (LABORATORY)	45
	6.a. Minimum Qualifications for Laboratory Analysts	45
	6.b. Calibration Procedures and Preventive Maintenance for Laboratory Instruments	47
	6.c. Analytical Methods	50
	6.d. Laboratory Quality Control Checks (blanks, dupli- cates, spike samples, and standards)	50
7	DATA REDUCTION, VALIDATION, AND REPORTING	59
	7.a. Standard Laboratory QA Forms	62
	7.b. Sampling Well Documentation	62
	7.c. Field Data Sheets	62

TABLE OF CONTENTS - Continued

<u>Section Number</u>	<u>Title</u>	<u>Page Number</u>
8	ROUTINE PROCEDURES FOR ASSESSING DATA PRECISION, ACCURACY, AND COMPLETENESS	63
	8.a. Precision Assessment	63
	8.b. Accuracy Assessment	63
	8.c. Completeness Assessment	64
	8.d. Detection Limit Calculation	64
	8.e. Quantitation Limit Calculations	64
9	PERFORMANCE AND SYSTEM AUDIIS	66
10	CORRECTIVE ACTION	67
11	QUALITY ASSURANCE REPORTS TO MANAGEMENT	68
	GLOSSARY OF TERMS	69
	REFERENCES	71
	APPENDIX	73

LIST OF FIGURES

<u>Figure Number</u>	<u>Title</u>	<u>Page Number</u>
3-1	Personnel responsible for quality assurance activities.	6
5-1	Suggested reporting form for field sampling including well purging and sample collection (modified from Barcelona, <u>et al.</u> , 1985a).	16
5-2	Sample alteration checklist.	17
5-3	Schematic of sample handling steps for ground water (from Barcelona, <u>et al.</u> , 1985).	35
5-4	Diagram of valving and sampling system enabling in-line filtration of water samples, and sequential switching of membrane filters if they clog (from Garske and Schock, 1986).	36a
6-1	Corrective action checklist.	60
9-1	Systems audit checklist.	76

LIST OF TABLES

<u>Table Number</u>	<u>Title</u>	<u>Page Number</u>
3-1	QA project organization.	7
3-2	QA officers responsible for laboratory and data analysis tasks.	9
4-1	Data quality objectives for water quality sampling by Ground Water Management Areas.	11
5-1	Sample container preparation, preservation, and handling for the most common ground water parameter groups.	13
5-2	Requirements for reagent water (from U.S. EPA, 1979a).	15
5-3	Example inorganic sample log (adapted from Barcelona, <u>et al.</u> , 1985a).	19
5-4	Example organic sample log (adapted from Barcelona, <u>et al.</u> , 1985a).	20
5-5	Calibration procedures, frequency, and preventive maintenance for field equipment.	21
5-6	Sampling mechanisms and performance ranking for monitoring wells (Barcelona, <u>et al.</u> , 1985a).	30
5-7	Recommendations for rigid materials in sampling applications (in decreasing order of preference) (Barcelona, <u>et al.</u> , 1985a).	32
5-8	Field quality control check samples.	40
5-9	Field standards and sample spiking solutions (from Barcelona, <u>et al.</u> , 1985a).	42
6-1	Skill-time rating of standard analytical operations (from EPA, 1979a).	46
6-2	Calibration procedures and frequency (adapted from Tetra Tech, Inc., 1986).	48
6-3	EPA methods and method detection limits for typical ground-water analytes.	52
6-4	Laboratory quality control check samples.	55

LIST OF TABLES - continued

<u>Table Number</u>	<u>Title</u>	<u>Page Number</u>
7-1	Recommended documentation for independent QA review of data on organic substances (from Tetra Tech, Inc., 1986).	61
7-2	Recommended documentation for independent QA review of data on inorganic substances (from Tetra Tech, Inc., 1986).	61a

Quality Assurance Interim Guidelines
for Water Quality Sampling and Analysis:
Ground Water Management Areas Program

1. INTRODUCTION

The Washington State Department of Ecology (Ecology) will administer and oversee grants to Ground Water Management Areas (GWMA) designated under Chapter 173-100 WAC with funds appropriated under Sections 4 and 9, Chapter 3, Title 70, Laws of 1986. Existing ground water quality must be characterized during development of a Ground Water Management Program, and in many areas, ground water sampling will be carried out. An on-going ground water monitoring system may also be part of each program's implementation plan. A comprehensive, well-documented Quality Assurance (QA) Project Plan is necessary to ensure that new data are of adequate quality for the purposes of the Ground Water Management Areas as well as for use by the Department of Ecology and other agencies.

QA procedures for ground water sample collection, analysis, and reporting are presented in this document. Many of these procedures are presented as guidelines rather than absolute requirements, recognizing the differences in ground water quality problems, monitoring goals, and priorities for problem resolution among the various GWMA's. The goals of this document are:

- To ensure that high-quality, verifiable data are collected.
- To encourage cost-effective use of resources.
- To ensure that data are usable by Ecology and other state and local agencies.

Specific activities that are covered in these guidelines include: project organization and responsibilities, objectives for data quality, methods for attaining these objectives including sampling and analytical procedures, and reporting requirements.

Each GWMA ground water sampling project will require a specific QA project plan. The essential elements for such a plan are included in this manual. While adjustments will be necessary to suit the specific objectives and situations unique to the individual project, each QA Project Plan must be organized in accordance with these guidelines (including the same 11 sections). All of the relevant issues and specific points in the guidelines must be included in the QA Project Plan. Justification must be provided for deviations from recommendations in these guidelines.

A draft QA Project Plan will be submitted to the Ecology Project Officer. Internal review of this draft plan by Ecology's technical review staff, followed by revisions negotiated between Ecology and the lead agency will result in a final QA Project Plan that will ensure that data collected through the GWMA projects will be accurate and comparable. Each QA Project Plan will be reviewed and approved by appropriate Ecology personnel before any samples are collected under the grant. As a project proceeds,

new or additional concerns may arise and adaptations to the QA Project Plan may be necessary to improve the project results. Periodic reports to the Lead Agency Project Manager and Ecology Project Officer referred to in Section 11 should bring attention to such issues. Consistency in methods of sampling, analysis, data evaluation, and reporting shall be a high priority in designing QA Project Plans. Careful and consistent review and tracking of project plans and deliverables is also critical. It is ultimately the responsibility of the Lead Agency Project Manager to ensure adherence to the final QA Project Plan.

All water quality samples, as a minimum, shall be analyzed for the following constituents unless otherwise approved by the Ecology Project Officer:

- temperature (field)
- pH (field)
- specific conductance (field)
- nitrate plus nitrite (laboratory)
- chloride (laboratory)
- iron (laboratory)

Additional constituents including major ions, trace metals, and synthetic organics, also may be analyzed based on the characteristics and history of a given GWMA and goals and objectives of the project.

It is assumed that three types of data may be used in characterizing the ground water in a GWMA: previously collected data, data collected by the GWMA from existing wells, and data collected by the GWMA from new monitoring wells. It is important to keep in mind that any data interpretation must take into account the reliability of the data (e.g., data with supporting information is more reliable than data without back-up). The following are minimum information requirements for data from wells to be used for GWMA ground water characterization:

1. Previously collected data - Well must have a Water Well Report Form that includes location information (down to quarter-quarter section) and well depth; sample data must indicate the name and/or agency of the person who collected the sample. Data must not be more than 20 years old.
2. Existing wells to be sampled as part of a GWMA - Same information as for #1, above. In addition, the Water Well Report Form must indicate the location and depth of the screened or open portion of the well, the casing material, and the elevation and horizontal location of the top of the casing must be surveyed by a licensed surveyor with reference to an established National Geodetic Datum. It is highly recommended that there be additional information associated with existing wells chosen for sampling such as the remaining items listed in Section 7.b. of these guidelines.
3. Monitoring wells to be drilled for data collection - Must provide same information as in 2., above. In addition, wells must meet the minimum information requirements listed in Ecology's "Design and Construction Guidelines for Monitoring Wells" and Section 7.b. of these guidelines.

The data forms for analytical results included in these guidelines must be used to report laboratory results. The field data forms provided in these guidelines (Figure 5-1 and Tables 5-3 and 5-4) also must be used. Similarly design forms that include at a minimum, the information required on the above-referenced forms, may be substituted.

2 PROJECT DESCRIPTION

The Ground Water Management Program's scope of work includes data collection and analysis in Phase III. This work is directed toward characterizing existing ground water conditions, including water quality. Acceptable existing data can be used to meet at least part of this need. Additional sampling will be necessary to a greater or lesser extent in most designated Ground water Management Areas depending on the spatial, temporal, and parametric coverage of available data.

Ground water sampling and analysis activities will require QA Project Plans for each GWMA. The purpose, goals, and objectives of the ground water sampling or monitoring effort should be described in the QA Project Plan. The specific sampling design can be included in the QA Project Plan or in a separate document. The sampling design will include site selection criteria, number of sites, parameters to be sampled, frequency of sampling, and data analysis methods. Rajagopal (1986) lists many references that address the various issues of ground water quality monitoring design and presents useful information regarding sampling frequency for characterizing ground water quality.

3. PROJECT ORGANIZATION AND RESPONSIBILITIES

Project organization and individuals responsible for quality assurance must be shown schematically in each QA Project Plan; for example as in Figure 3-1. Specific responsibilities of these individuals also will be summarized in table format (Table 3-1 as an example). QA officers charged with tracking specific laboratory and data analysis tasks also should be shown in a table such as Table 3-2.

Specific time requirements should be made in the QA Project Plan for submission of data deliverables. Responsibility for meeting these time schedules should be assigned to the Contractor Project Manager or someone at that level of organization with oversight responsibilities by the Lead Agency Project Manager. Penalties for lateness should be part of any laboratory or consultant contract, with stipulations for certain extenuating circumstance. Tetra Tech (1985c) includes guidelines for developing and using a time chart to keep track of project and QA activities.

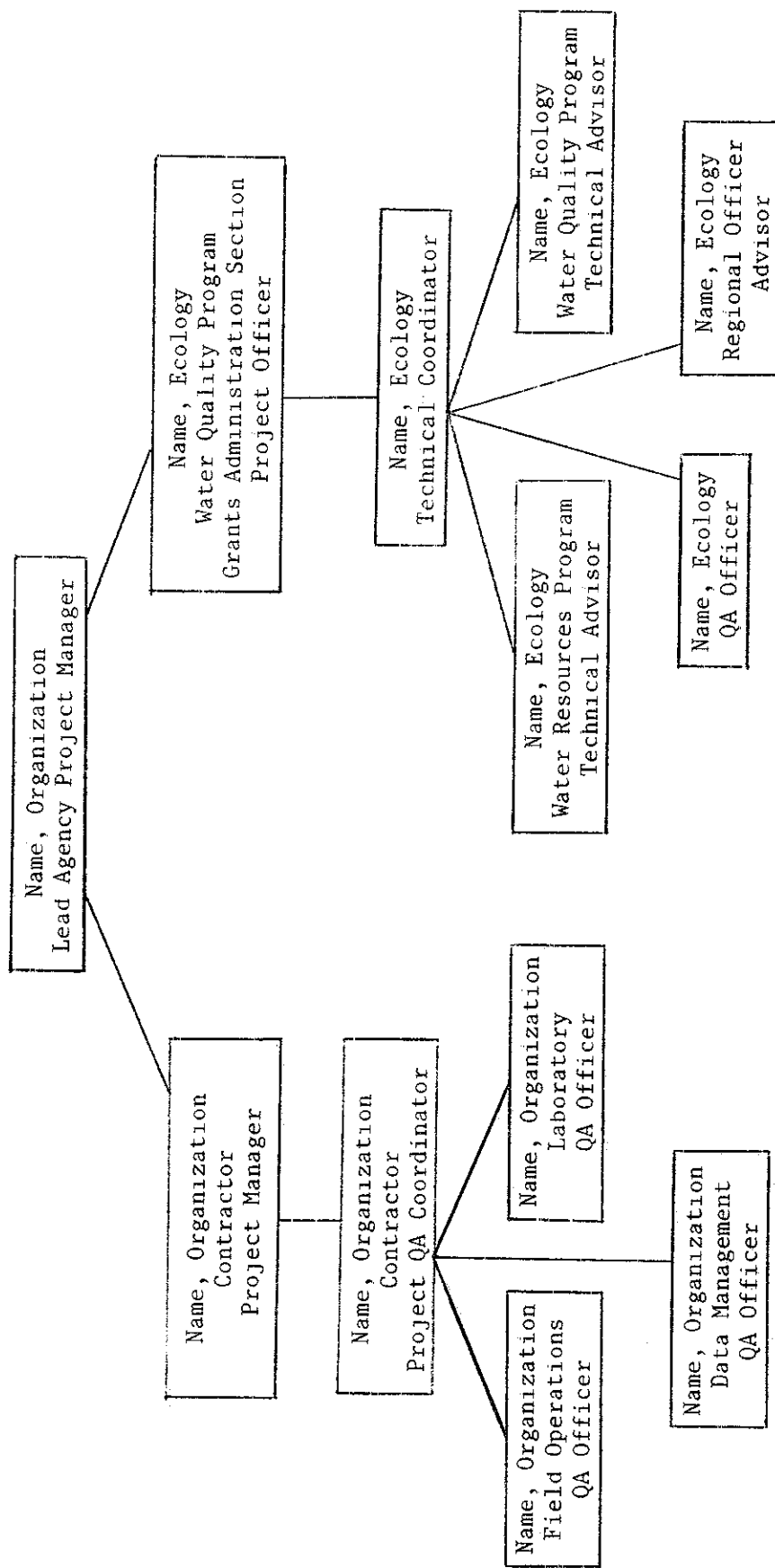


Figure 3-1. Hypothetical QA project organization.

Table 3-1. Personnel responsible for quality assurance activities.

<u>Personnel</u>	<u>Responsibilities</u>
Ecology Grants Administration Section Supervisor	Consults with Ecology Project Officer on policy decisions and major QA protocol problems.
Ecology Project Officer	Lead person in Ecology responsible for project performance, review of project QA needs and problems, and approval of appropriate QA corrective actions as needed.
Ecology QA Officer	Provide technical QA assistance in relation to state policies, regulations, and procedures.
Ecology Technical Coordinator	Responsible to Ecology's Project Officer for providing technical review on grant application, QA plans and data.
Ecology Technical Advisor	Assists Ecology Technical Coordinator and QA Officer in reviewing QA Project Plans and data submitted. Also may assist in tracking fulfillment of WA Project Plan provisions.
Lead Agency Project Manager	Lead agency responsibilities for review and approval of QA Project Plan. Responsible for ensuring that QA Project Plan is carried out to the full extent and that data collected meet the specified data quality objectives.
Contractor Project Manager	Oversees field, laboratory, and data analysis, and QA activities to ensure that project performance complies with QA Project Plan. Acts as liaison between agencies and contract personnel. Approves and implements necessary corrective action and adjustments to accomplish project objectives. Promptly notifies GWMA project manager and Ecology Project Officer of any corrective action or adjustments made to schedules or procedures. Submits QA results with sampling data and other documentation as described under Sections 5, 7, and 11 on a regular basis (depending on the project schedule).

Table 3-1 continued.

<u>Personnel</u>	<u>Responsibilities</u>
Contractor Project QA Coordinator	Oversees all contractor QA activities to ensure compliance with QA Project Plan specifications. Coordinates corrective actions. Responsible for tracking project results of field, laboratory, and data management QA activities.
Field Operations QA Officer (Contractor)	Tracks QA activities relative to field sampling and analysis to ensure compliance with QA Project Plan. Conducts inspections of sampling procedures, field analyses, and documentation in conjunction with QA Project Plan specifications. Verifies that laboratory results of field blanks, duplicates, spikes, and standard reference materials are being used to evaluate and eliminate any potential sample contamination. If problems are discovered, reports these immediately to Contractor Project QA Coordinator.
Laboratory QA Officer (Contractor)	Tracks QA activities and sample results from the laboratory. Evaluates laboratory adherence to procedures required in the QA Project Plan. When problems discovered, reports these immediately to Contractor Project QA Coordinator. Submits reports to Contractor Project QA Coordinator on a specified time basis (time frame dependent on project schedule).
Data Management QA Officer (Contractor)	Reviews all project QA documentation to ensure that procedures carried out by field, laboratory, and other project personnel comply with QA Project Plan objectives and requirements. Submits QA reports to Contractor Project QA Coordinator on a specified time basis (time frame dependent on project schedule.)

Table 3-2. QA officers responsible for laboratory and data analysis quality assurance.

<u>QA Officers</u>	<u>Responsibility</u>
Name, Organization	Organic compounds
Name, Organization	Metals
Name, Organization	Ancillary parameters
Name, Organization	Data analysis

4. OBJECTIVES FOR MEASUREMENT DATA

The objectives of the QA process are to develop and carry out relatively standardized procedures for field sampling, laboratory analysis, and reporting that will provide reliable scientific data useful for the individual Ground Water Management Areas as well as other data users. Specific procedures to be used for sample collection, field analysis, laboratory analysis, quality control checks, preventive maintenance, reporting, and corrective actions are described in other sections of these QA Guidelines.

The purpose of this section is to define recommended QA goals for detection limits, accuracy, precision, and completeness. These goals are shown in Table 4-1. Such a table is an essential part of each QA Project Plan, since it presents the acceptable standards that field and laboratory teams must plan to meet before sampling begins. After sampling and analysis, data and project results are again compared to these goals to determine if they actually meet the goals.

Definitions of accuracy, precision, completeness, and detection limits are presented in Section 8 and in the Glossary of Terms at the end of this report. These measures are evaluated by analysis of spiked samples, duplicates, standard reference materials, and blanks. Recommended procedures and frequencies for these samples are described in 5.i. and 6.d.5.

Table 4-1. Data quality objectives for water quality sampling by Ground Water Management Areas.

Variable ^a	Matrix	Units	Lower Limit of Detection	Accuracy ^b	Precision ^b	Completeness ^b	Preferred Method ^c	Reference	Maximum Holding Time ^d
ORGANICS									
Volatile organics	Water	ug/L	nd 7.2 ^e	± 20%	± 20%	95%	EPA Method 624	Federal Register, 40 CFR Part 136	14 days.
Base neutrals and acids	Water	ug/L	0.9-42 ^e	± 20%	± 20%	95%	EPA Method 625	Federal Register, 40 CFR Part 136	7 days/40 days
Organochlorine pesticides and PCBs	Water	ug/L	0.003-0.24 ^e	± 20%	± 20%	95%	EPA Method 608	Federal Register, 40 CFR Part 136	7 days/40 days
Purgable halocarbons	Water	ug/L	0.03-1.81 ^e	± 20%	± 20%	95%	EPA Method 601	Federal Register, 40 CFR Part 136	7 days/40 days
Polynuclear aromatic hydrocarbons	Water	ug/L	0.13-2.3 ^e	± 20%	± 20%	95%	EPA Method 610	Federal Register, 40 CFR Part 136	7 days/40 days
INORGANICS AND CONVENTIONALS									
Trace elements (metals such as Al, As, Ba, Cd, Cr(VI), Fe, Mn, Pb, Hg, Se, Zn)	Water	ug/L	e	± 20%	± 20%	95%	EPA Methods, g 1979	U.S. EPA, 1979	Depends on the metal. See Table 6-3.
Total organic carbon	Water	ug/L	0.5	± 10%	± 10%	95%	EPA Method 415.1	U.S. EPA, 1979	24 hrs.
Nutrients and inorganics (Such as Ca, Na, K, Si, NO ₃ , NO ₂ , P, dissolved solids, SO ₄ , Cl, F, HCO ₃ , CO ₃)	Water	ug/L (mg/L for concentrations greater than 1.0 mg/L)	e	± 10%	± 10%	95%	EPA Methods, g, h 1979	U.S. EPA, 1979	Depends on constituents. See Table 6-3.
pH	Water	Standard pH units	N/A	± 0.1 pH units	± 0.1 pH units	95%	EPA Method 150.1	U.S. EPA, 1979	Analyze immediately.
Specific conductance	Water	umhos/cm	1.0	± 5%	± 5%	95%	EPA Method 120.1	U.S. EPA, 1979	24 hrs.
Eh	Water	Volts	N/A	± 20%	± 20%	95% (where D.O. less than 0.01 mg/L and hydrogen sulfide not present)	USGS, 1980 ⁱ	USGS, 1980	Analyze immediately before sample exposed to the atmosphere.
Dissolved oxygen	Water	mg/L	0.1	± 0.2 mg/L	± 0.2 mg/L	95%	EPA Method 360.2	U.S. EPA, 1979	Fix samples immediately in the field; analyze within 4-8 hours.
Hardness	Water	mg/L				95%	EPA Method 130.2	U.S. EPA, 1979	Immediately or as soon as possible (24 hr. maximum).
Alkalinity	Water	mg/L CaCO ₃	1.0	± 10%	± 5%	95%	EPA Method 310.1	U.S. EPA, 1979	Measure static water level before any water removed.
Water level	Water	ft		± 0.01 ft ^j	± 0.01 ft	99%	USGS, 1980 ⁱ	USGS, 1980	Measure immediately.
Temperature	Water	degrees C	4°C	± 0.1 °C	± 0.1 °C	99%	EPA Method 170.1 or using temperature electrode	U.S. EPA, 1979 (or manufacturer's recommendations for electrode)	

Footnotes:

- nd - not determined.
a - See Table 6-3 for additional possible variables, accepted methods, and holding times.
b - See Section 8 for techniques used to calculate.
c - See Table 6-3 for other acceptable methods depending on the study objectives.
d - Where two times are given, the first refers to the maximum time prior to extraction, the second to the maximum time prior to instrumental analysis.
e - Depends on the individual compound. Lower limit of detection must not exceed the method detection limit.
f - Depends on the individual compound. Accuracy is determined by spike recoveries and standard reference materials. Acceptance criteria for results of these analyses given in the method serve as accuracy limits.
g - U.S. EPA, 1979. Methods for chemical analysis of water and wastes. EPA-600/4-79-020. Two methods are given for most metals. The atomic absorption furnace technique is recommended, since the method detection limits are much lower than for the direct aspiration technique.
h - If no method provided for constituent of interest, see Federal Register 40 CFR Part 136 (Table 1B) for other EPA-approved methods.
i - U.S. Geological Survey, 1980. National handbook of recommended methods for water-data acquisition, Reston, VI. Chapter 2.
j - If E-Cape is used, accuracy can be measured in comparison with steel tape measurement.
k - Federal Register, October 24, 1984, 40 CFR Part 136. Guidelines Establishing Test Procedures for the Analysis of Pollutants under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule.

AV/MQ3/86/120201A

5. SAMPLING AND FIELD MEASUREMENT

All personnel involved in sampling and field analysis must be familiar with the QA Project Plan developed for that GWMA sampling and laboratory effort, especially the areas dealing with sampling and field measurement.

5.a. Sample Container Preparation and Preservatives

Table 5-1 presents accepted container preparation procedures, preservation methods, and limited sample handling information for the major categories of ground water analytes. Variations from these general recommendations are necessary for certain specific constituents, and should be specified in each quality assurance project plan, preferably in a similar table format.

Containers should be sealed and stored in a clean environment after dry and cool to prevent contamination. Even new bottles should be cleaned according to these procedures. For inorganics and non-volatile organics if preservatives are added in the field (preferred), rinse the sample container with sample water first. If preservatives are added to containers before going in the field, DO NOT pre-rinse with sample.

Minimum requirements for final rinse water for different containers (Types I - IV) are shown on Table 5-2.

Deionized, distilled water is to be used for the final container rinses on containers to be used for metals samples.

5.b. Field Notes

A notebook should be kept and used in the field for documenting information relevant to sample collection at the time the samples are collected. Notes should be taken using indelible ink, kept in a ring binder, or bound together in some way. Figure 5-1 should be part of the notebook and filled out completely for each sampling site (e.g., well). Measurements for temperature, pH, specific conductance, and Eh should be recorded periodically while the well is purging (e.g., at least once for each well bore purged) and after all samples are collected. Results of all other field analyses should be recorded in the field notebook. Information related to the well's recent pumping history should be recorded if the well is in use. It is often helpful to bring copies of the well logs and field notes recorded during previous samplings.

Any deviation from the standard protocol should be noted and, if relevant, communicated to the lab with the samples. The Sample Alteration Checklist (Figure 5-2) should also be used to notify the Ecology Project Officer (through the Contractor Project Manager). However, telephone communication is also recommended for severe problems.

Before sampling, the outside of the well should be inspected. If there are any signs of problems such as cracking of the seal or vandalism, note this in the field notebook.

Table 5-1. Sample container preparation, preservation, and handling for the most common ground-water parameter groups.

<u>Parameter Group</u>	<u>Container</u>	<u>Container Preparation</u>	<u>Sample Preservation and Handling</u>
Volatile Organics	Two 40-mL glass vials w/teflon-lined septum cap (preferable amber colored vials)	Wash with non-phosphate detergent in hot tap water (vials, teflon liners, and caps). Rinse 3 times w/ distilled water followed by 3 deionized water rinses (Type I or purer water). Oven dry vials, septa, and caps at 105°C for more than 1 hour. Wearing gloves, place septa inside cap, teflon side down, and screw onto vial.	Fill <u>slowly</u> leaving <u>no</u> air-space. If even a small air bubble is visible when vial is inverted, sample must be collected again. Keep in dark on ice (4°C)
Acid/Base Neutrals -Priority Pollutants	Two 1/2-gal, one 1-gal or four 1-liter amber glass bottle(s) w/teflon-lined septum cap(s)	Wash with non-phosphate detergent in hot tap water (bottles, teflon liners, and caps). Rinse 3 times w/tap water. Rinse with 50% nitric acid. Rinse 3 times w/de-ionized water (Type I or purer water). Rinse w/pesticide grade methylene chloride or hexane. Oven dry. Wearing gloves, place teflon liners in caps teflon-side down and screw into vial.	Leave headspace. Keep in dark on ice (4°C)
Trace Metals	High-density linear polyethylene bottles	Wash with non-phosphate detergent in hot tap water (bottles and lids). Rinse 3 times w/hot tap water. Soak in 20% nitric acid. Rinse at least 5 times w/de-ionized water (Type I or purer water). Air dry in contaminant-free environment. Screw lids loosely on bottles.	Total metals - add distilled nitric acid for trace metals analysis to pH <2. Dissolved metals - filter, then same as total metals.
Major Anions and Cations	High-density linear polyethylene bottles	Wash with non-phosphate detergent in hot tap water (bottles and lids). Rinse w/ distilled water. Rinse w/20% nitric acid. Rinse 5 times w/de-ionized water (Type II or purer water). Air dry in contaminant-free environment. Screw lids loosely on bottles.	Add 1 mL concentrated nitric acid/liter; or to pH <2

Table 5-1 continued.

<u>Parameter Group</u>	<u>Container</u>	<u>Container Preparation</u>	<u>Sample Preservation and Handling</u>
Ammonia, Nitrate, nitrite, nitrate+nitrite	High-density linear polyethylene bottles	Same as major anions and cations.	Ammonia - analyze as soon as possible or add sulfuric acid to pH <2, refrigerate (4°C). Nitrate - add sulfuric acid to pH <2, refrigerate (4°C). Nitrite - analyze as soon as possible or refrigerate; or freeze at -20 °C. Nitrate+nitrite - same as nitrite.
pH, alkalinity	Same as above	Prewash containers and caps w/hot tap water. Wash with non-phosphate detergent and hot tap water. Rinse with hot tap water. Rinse w/distilled water (Type II or purer water). Air dry and screw caps on containers.	Analyze immediately in field. If impossible, keep in dark on ice (4°C).
Hardness, Specific Conductance	Same as above.	Same as above.	Keep on ice (4°C). Hardness - add nitric acid to pH <2.
Dissolved Solids	Save as above.	Same as above.	Filter on site. Keep in dark on ice (4°C).

References:

1. Ecology Sampling Containers Cleaning and Preservation Protocols (1986).
2. APHA, AWWA, WPCF, 1986. Standard Methods for the Examination of Water and Wastewater.
3. Tetra Tech, Inc., 1986. Quality Assurance Project Plan for Field Investigations to Support Commencement Bay Nearshore/Tideflats Feasibility Study. Prepared for Washington State Department of Ecology.
4. Golder Associates, Inc., April 1986. Quality Assurance Project Plan for Remedial Investigations at the North Market Street Site. Prepared for the Washington State Department of Ecology.
5. U.S. EPA, 1979. Methods for Chemical Analysis of Water and Waste. EPA-600/4-79-020.
6. Univ. of Waterloo, Earth Science Group, 1980. Field Methods in Contaminated Hydrogeology.
7. U.S. EPA, 1979. Handbook of Analytical Quality Control in Water and Wastewater Laboratories, EPA-600/4-79-019.

Table 5-2. Requirements for reagent water (from U.S. EPA, 1979a).

Grade of Water	Maximum Total Matter (mg/L)	Maximum Electrical Conductivity at 25° C (μ mho/cm)	Minimum Electrical Resistivity* at 25° C (M Ω • cm)	pH at 25° C	Minimum Color Retention Time of KmnO_4 (min)
Type I	0.1	0.06	16.67	-	60
Type II	0.1	1.0	1.0	-	60
Type III	1.0	1.0	1.0	6.2-7.5	10
Type IV	2.0	5.0	0.2	5.0-8.0	10

Figure 5-1. Suggested reporting form for field sampling including well purging and sample collection (modified from Barcelona, et al., 1985a).

GROUND-WATER SAMPLING RECORD

[illegible]

* $V = 3.14 r^2 \times h$, where V = volume of water in casing (cubic feet), r = radius of the casing (feet), and h = length of casing filled with water (feet) (1 cubic foot = 7.48 gallons).

SAMPLE ALTERATION CHECKLIST

Project Name and Number:

Material to be sampled:

Measurement Parameter:

Standard Procedure for Field Collection & Laboratory Analysis (cite references):

Reason for change in Field Procedure or Analytical Variation:

Variation from Field or Analytical Procedure:

Special Equipment, Materials or Personnel Required:

Initiators Name: _____ Date: _____

Project Approval: _____ Date: _____

Laboratory Approval: _____ Date: _____

QA Officer/Reviewer: _____ Date: _____

Figure 5-2. Sample alteration checklist.

Tables 5-3 and 5-4 also should be part of the field notebook and filled out completely at the sampling site. At least half of the field blanks, standards, and spikes should not be labelled for the laboratory as such. These are to be treated the same as any other sample since they are intended to indicate the effect of normal sample treatment on the reliability of results. The other half of the field blanks, standards, and spikes should be labeled as such to aid the analyst in detecting and evaluating problems. Sample numbering is one way of disguising blank, split, or duplicate samples. The sample form (Figure 5-1) is the key to the actual collection location or identity as blank, duplicate, etc.

Any other pertinent or unusual information should also be recorded in the field notebook, such as sample color or odor, and time of calibration for field instruments, and method of calibration (if not standard). Field notes should be kept by the contractor and/or copies sent to the Lead Agency Project Manager. Results of field analyses, data forms (Figure 5-1; Tables 5-3 and 5-4), and other pertinent information must be included in data reports to the Ecology Project Officer as described in Section 11.

5.c. Calibration Procedures and Frequency for Field Instruments and Preventive Maintenance

Specific equipment to be used for field analyses should be listed in the QA Project Plan, including the name of the manufacturer and the model number. Routine preventive maintenance procedures should also be specified for each instrument.

Calibration procedures and frequency of calibration shall be recorded in appropriate log book(s) and shall represent accepted techniques to ensure accurate sampling, testing, and documentation. All equipment shall be, as a minimum, calibrated per the manufacturer's directions.

A suggested format for documenting calibration procedures and frequency for each field variable and preventive maintenance is shown in Table 5-5 (from Golder and Associates, 1986).

5.d. Design and Construction Guidelines for Monitoring Wells

New wells drilled as part of the GWMA's sampling or monitoring programs must follow the Ecology "Design and Construction Guidelines for Monitoring Wells" available through the Water Quality Program. Documentation of drilling details should be submitted to the Ecology Project Officer through the Lead Agency Project Manager. At a minimum, items listed under 7.b. must be submitted.

5.e. Static Water Level Measurement (USGS, 1980; NCASI, 1982)

Each GWMA QA Project Plan should include the standard protocol to be used for measuring static water level in sampling wells. The following are examples and recommended procedures.

Table 5-3. Example inorganic sample log (filtered samples if required) (adapted from Barcelona, et al., 1985a).

Date 1/1/84 (time)	Well Number	Sample Number	Alka- linity (field) Volume (mL) #	Major Anions (HDPE) Volume (L) #	Major Cations (HDPE) Volume (L) #	Trace Metal Ions (HDPE) Volume	Non- metallic Inorganics (HPDE) Volume
	1	1A	X				
a.m.		1B		X			
p.m.		1C			X		
		1D				X	
		1E					X
	2	2A	X				
a.m.		2B		X			
p.m.		2C			X		
		2D				X	
		2E					X
	3	3A	X				
a.m.		3B		X			
p.m.		3C			X		
		3D				X	
		3E					X
	3*	5A	X				
a.m.		5B		X			
p.m.		5C			X		
		5D				X	
		5E					X
	4	4A	X				
a.m.		4B		X			
p.m.		4C			X		
		4D				X	
		4E					X
			Conc (ppm)	Conc (ppm)	Conc (ppm)	Conc (ppb)	Conc (ppb)
Field Blanks		6A	0				
a.m.		6B		0			
p.m.		6C			0		
		6D				0	
		6E					0
Field Stds.		7A	100				
a.m.		7B		25			
p.m.		7C			50		
		7D				50	
		7E					100
Split Sample		4A-SS	100				
Spikes		4B-SS		25			
		4C-SS			50		
		4D-SS				50	
		4E-SS					150

*Example of duplicate labeling so that the laboratory is not aware that two samples are duplicates. Sample numbers for blanks and standards need not follow real sample numbers. Variety in spiking levels and standards is encouraged (within the expected range for GWMA samples) so that laboratory analysis procedures are truly being tested.

Table 5-4. Example organic sample log (lab filtered, if necessary)
(adapted from Barcelona, et al., 1985a).

Date (time)	Well Number	Sample Number	IOC 40 mL	TOX amber glass vials	Volatiles	Extractables	
						A FFE sealed amber glass Volume (L)	B (etc.) Volume (L)
	1	1A0	X				
a.m.		1B0		X			
p.m.		1C0			X		
		1D0				X	
		1E0					X
	2	2A0	X				
a.m.		2B0		X			
p.m.		2C0			X		
		2D0				X	
		2E0					X
	2*	5A0	X				
a.m.		5B0		X			
p.m.		5C0			X		
		5D0				X	
		5E0					X
	3	3A0	X				
a.m.		3B0		X			
p.m.		3C0			X		
		3D0				X	
		3E0					X
	4	4A0	X				
a.m.		4B0		X			
p.m.		4C0			X		
		4D0				X	
		4E0					X
			Conc. (mg/L)	Conc. (ug/L)	Conc. (ug/L)	Conc. (ug/L)	Conc. (ug/L)
Field Blank		6A0	0				
a.m.		6B0		0			
p.m.		6C0			0		
		6D0				0	
		6E0					0
Field Stds.		7A0	2				
a.m.		7B0		25			
p.m.		7C0			25		
		7D0				50	
		7E0					25
Split Sample		4A0-SS-1	2				
Spikes		4B0-SS-1		25			
a.m.		4C0-SS-1			25		
p.m.		4D0-SS-1				25	
		4E0-SS-1					25

*Example of duplicate labeling so that the laboratory is not aware that two samples are duplicates. Sample numbers for blanks and standards need not follow real sample numbers. Variety in spiking levels and standards is encouraged (within the expected range for samples) so that laboratory analysis procedures are truly being tested.

Table 5-5. Calibration procedures, frequency, and preventive maintenance for field equipment.

<u>Equipment and Accessories</u>	<u>Calibration</u>	<u>Frequency (Minimum)</u>	<u>Preventive Maintenance</u>
<u>Water-Level Measuring Devices</u>			
Electric Water-Level Tape	Calibrate with steel surveyor's tape. Before sampling, probe can be dipped in water to check battery.	Once every month at each well.	Pre-survey check. Bring extra batteries.
Steel surveyor's tape	Calibrate using manufacturer-supplied temperature correction if applicable for field conditions.	At each well.	Pre-survey check. Bring extra weights.
Thermometers - mercury or alcohol	Calibrated once, check reading to make sure no breaks in alcohol reservoir or measurement line.	Check for fluid breaks before each new station reading.	If sampling for mercury, do not use mercury thermometer. If thermometer breaks, samples will likely be contaminated. Bring extras.
Temperature probe	Calibrate with thermometer.	Calibrate before each new station.	
pH meters	Calibrate electrode to pH 4 and pH 7 buffer solutions (or to span expected range of sample). Buffers, electrode, and samples must be same temperature. Allow 10-15 minutes for equilibration, bathing electrodes and buffers with purge water before sample collection (if temperatures of instrument and buffers are different from sample water). (See USGS, 1980, for more details.)	Calibrate before use at each sample site.	Pre-survey check; spare pH probes; spare buffer solutions. New buffers should be made up or obtained from the factory at least once every 6 months.

Table 5-5 continued.

<u>Equipment and Accessories</u>	<u>Calibration</u>	<u>Frequency (Minimum)</u>	<u>Preventive Maintenance</u>
Specific conductance meters	"Red line" calibration on the YSI Model 33 conductivity meter. Temperature correction applied during measurement.	Calibrate before each reading.	Pre-survey check, spare probes; spare probe cable.
	Test with Standard KCL Solution - fresh every 6 months (minimum of 2 solutions to cover range of meter).	Daily	
	Factory calibration (see USGS, 1980, for more details).	Annually	
Dissolved oxygen meter (if used)	Calibrate as specified in instruction manual (compare to Winkler Method - at least one sample at beginning of day and one at end of day). If interferences are suspected, calibrate w/distilled water aerated to saturation (USGS, 1980).	Daily - at the beginning and end of sampling day.	
Eh redox-potential meters	Test with Zobell (USGS, 1980) (Zobell solution should be equilibrated to temperature of sample. Allow 10-15 min. to reach equilibration.)	Calibrate at the beginning and end of sampling day.	Pre-survey check; spare probes; reagent-grade quinhydrone.
Pressure transducers	Factory calibration and periodically by contractor using water columns.	Every 6 months. Before each set of water level measurements.	

Table 5-5 continued.

<u>Equipment and Accessories</u>	<u>Calibration</u>	<u>Frequency (Minimum)</u>	<u>Preventive Maintenance</u>
Sample containers and preservatives.	N/A	N/A	Pre-survey check; spare containers, coolers, preservatives.
Compressed gas bottled	N/A	N/A	Pre-survey check; spare gas bottles; spare wrenches.
Sampling pump	Check flow rate using volumetric measuring vessel and stopwatch.	Before going into field; before sampling first site; periodically during sampling day.	Pre-survey check; spare parts, tools.
Sampling tubing	N/A	N/A	Pre-survey check; spare tubing.
Distilled or deionized, distilled water	N/A	N/A	Extra distilled or deionized, distilled water.
Graduated buckets	Check volume with calibrated measuring vessels.	One time before sampling.	Spare buckets.

Rinse probes and electrodes with distilled water (squeeze bottle) after all measurements. Keep meters out of direct sunlight and off wet surfaces.

The non-pumping level of water in the well should be measured at each well before any water is removed from the borehole. The elevation of a marked point at the top of the well casing must be surveyed by a licensed surveyor prior to sampling with reference to an established National Geodetic Datum (USGS datum).

The following procedure is recommended for static water level measurement:

1. Record well identification information in field notebook (Figure 5-1).
2. If cleanliness of downhole equipment is in question, pre-clean in field prior to use. Clean downhole electrical water level measuring tape or graduated steel tape between wells, especially if previous samples have indicated contamination.

Graduated Steel Tape Method (USGS, 1980). This technique is not recommended for wells of 2-inch diameter or less, as weight and tape may raise water level somewhat.

- Attach a narrow weight to the end of the steel measuring tape with wire strong enough to hold the weight but not as strong as the tape.
- Chalk the lower few feet of the tape by pulling it across a piece of carpenter's chalk.
- Slowly lower tape to avoid splashing.
- When weight and tape are felt to be submerged in water, record the tape measurement at the top of the well casing.
- Pull tape to the surface and record the tape measurement at the water mark.
- Subtract the measurement at the water mark from that at the top of the casing to obtain the depth to water and record it in the field log.
- Repeat the above procedure at least once more. The second measurement should be taken and recorded several minutes after the initial reading. If the two readings do not agree within ± 0.01 to 0.02 feet, continue to take measurements until consistent readings are obtained.

Electrical Tape Method (USGS, 1980)

- Electrical tape should be marked off in 5-foot intervals with metal bands or color-coded markers.
- Lower probe into the well slowly until the instrument indicates contact with water.

- Mark tape at the top of the well casing and withdraw to first metal measuring band.
- Record distance from the mark to the metal band with a measuring tape or folding rule. Add that distance to the reading at the metal band to obtain the depth to water, and record.
- Tape should not rub against the top of the well casing as metal bands can be moved.
- Repeat procedure at least once. Second measurement should be taken and recorded several minutes after initial reading. If the two readings do not agree within ± 0.04 foot, continue to take measurements until consistent readings are obtained.

(It should be kept in mind that an electrical tape will not show a reading for an oil layer floating on the water surface unless special adaptations are made to the tape.)

At least the first time a well is used for sampling the depth to the bottom of the well should be measured and recorded to detect any problems with well collapse or objects deposited in the well.

5.f. Well Purging Prior to Sample Collection

Detailed standard procedures for purging stagnant water in sampling wells should be included in each QA Project Plan. The following recommended procedures and protocols may be used and modifications specified as appropriate in the QA Project Plan.

- Exercise caution in purging to prevent introduction of contaminants from around the well or from other wells.
- Principle: Remove water standing in casing, storage tanks, or piping which may not be representative of aquifer water.
- Document details of purging process in field notes (Figure 5-1 and additional notes as necessary).

5.f.1. For Monitoring Wells:

- Start sampling with least contaminated well; work toward most contaminated.
- Place a dropcloth on the ground around the well and work area to prevent introduction of contaminants into the well, sampling equipment, and/or samples.
- Use a pump that will not introduce contaminants into the well and can be cleaned between wells. (Oil and grease are often a problem with submersibles.) Preferred purging equipment in order of preference is: teflon bladder pump; stainless steel bladder pump;

teflon or stainless steel dual check valve bailer; mechanical positive displacement pump (Barcelona, et al., 1985a).

- Purge water should be disposed of so that it does not directly re-enter or affect the well. Purge water should NEVER be poured or pumped back into the well.
- Purge equipment needed for specially constructed monitoring wells should be thoroughly cleaned before use. Submersible pumps that allow well water to contact oil, grease, metal, or rubber parts should be avoided even if metals and organics are not being sampled as part of the GWMA sampling project. These constituents may be sampled in the future, and the well may be suspect if such pumps have been used in the past. A suggested procedure for cleaning purging equipment, especially if the same equipment is to be used for sampling, is to rinse in dilute hydrochloric acid, followed by several rinses with tap water and distilled or de-ionized water. If organics are of interest, one rinse with acetone, hexane, or methanol should be added, and the equipment allowed to dry before using, to prevent contamination of the well (Barcelona, et al., 1985a).
- Measure static water level as recommended above before beginning to purge. Calculate storage volume of the well bore as:

$$\text{Volume (V)} = 3.14 \times R^2 \times L \quad (1)$$

V = Borehole volume (cubic feet)

R = Radius of well bore (feet)

L = Length of static water in well bore (feet)

(1 cubic foot = 7.48 gallons)

If transmissivity data are available for the immediate area, follow protocol A. If transmissivity data lacking, follow protocol B.

5.f.1.1. Protocol A

- Use the Papadopulus and Cooper equations (Papadopulus and Cooper, 1967) as guidelines to calculate length of time the well should be pumped at a given rate until well contains about 90 percent aquifer water (or volume removed if purging with a bailer) (Barcelona, et al., 1985a). This takes into consideration the amount of casing water being pumped.
- Pump intake should be as close to the water surface as possible (within a few inches).
- Measure water level periodically during purging. Sufficient water should always be left in the well to cover the screened section of the well. (Try NOT to pump well to dryness as this may allow gases to be exchanged with the atmosphere which can affect constituents being sampled.)

- Appropriate pumping rate depends on the size of the well and hydraulic conductivity of the formation. Choose a pumping rate that minimizes drawdown and pumping time (500 mL/min. recommended for small-diameter wells -- Barcelona, et al., 1985a).
- Note pumping rate and any changes to rate and time in field notes (e.g., Figure 5-1).
- Periodically measure the water level during purging (at least once for each well volume).
- If possible, monitor purge water with in-line, flow-through device for indicator parameters (temperature, pH, specific conductance, and Eh). Record readings at least once for each well volume purged on Figure 5-1 (Garske and Schock, 1986; Barcelona, et al., 1985a; USGS, 1980; NCASI, 1982). Record readings just prior to sample collection. This information is helpful in evaluating reliability of transmissivity assumptions and changes in sample water properties. When readings for indicator parameters vary less than +10 percent over two consecutive bore volumes, this is a good indication that you are sampling mostly aquifer water (Barcelona, et al., 1985a).
- If an in-line, flow-through system is not used, measure and record temperature, pH, and specific conductance several times during purging from discharge water according to USGS (1980).
- Sampling equipment should also be completely purged after cleaning with sample water before collecting samples.

5.f.1.2. Protocol B

- With pump intake as close to the water surface as possible (within a few inches), pump at least 5 bore volumes if well is productive enough (NCASI, 1982). Sufficient water should always be left in the well to cover screened section. (If circumstances do not allow 5 bore volumes to be purged, an absolute minimum of 1 bore volume must be purged.)
- Measure water level periodically during purging and record on Figure 5-1.
- Appropriate purging rate depends on size of the well and hydraulic conductivity (or transmissivity). Choose a pumping rate that minimizes drawdown and time spent (500 mL/min. recommended for small-diameter wells by Barcelona, et al., 1985a). Record purging rate on field sheet (e.g., Figure 5-1).
- If possible, monitor purge water with in-line, flow-through device for indicator parameters (temperature, pH, specific conductance, Eh), and record readings periodically on Figure 5-1 (Garske and Schock, 1986; Barcelona, et al., 1985a; USGS, 1980;

NCASI, 1982). This information is helpful in evaluating whether five bore volumes are adequate to guarantee that aquifer water is being sampled. When readings for indicator parameters vary less than ± 10 percent over two consecutive bore volumes, it is a good indication that mostly aquifer water is being obtained (Barcelona, et al., 1985a).

- Sampling equipment should also be completely purged with sample water after cleaning and before collecting samples for laboratory analysis.

5 f.2. For Non-Monitoring Wells (wells with installed pumps)

- If possible, determine recent pumping schedule for the particular well to be sampled and nearby wells if part of a wellfield. Sampling well (and preferably any nearby wells) should not have been pumped for at least two hours before sampling.
- Measure depth to static water as described previously, if possible. Be sure that pump is not used while measuring devices are in the well.
- If transmissivity information is available for the location, use the Papadopolus and Cooper (1967) equations, as recommended under Protocol A, above.
- If transmissivity information is not available, using well dimensions and depth to static water, calculate well storage volume (see equation 1, above). Add volume of any storage tank(s) and lines through which sample water will travel before collection to obtain total volume of stagnant water. (If static water level measurement is not possible, estimate the amount of static water in the well and explain in field notes. Conservative estimate is recommended; e.g., assume water level at surface.)
- Pump at least three storage volumes (well bore plus storage tanks and pipe(s) if in the line of flow to sampling [and purging] point).
- The slower the pumping rate during purging, the better. Since time usually is a constraint, a suggested rate for large supply wells is 20 to 50 gal/min. (Schmidt, 1983). Smaller wells should be pumped at much slower rates.
- Allow water to flow into a bucket or catch basin and monitor indicator parameters (temperature, pH, specific conductance) during purging. (Eh values will not be meaningful if the sample is exposed to the air.) Record periodically in field notes (Figure 1). After three storage volumes are purged, if indicator parameters vary more than ± 10 percent over a few minutes, continue to purge until less than 10 percent.

- Measure and record purging rate; e.g., using five-gallon bucket and stopwatch. Record in field notes (Figure 5-1).

5.g. Sampling Mechanics

5.g.1. Sampling Mechanisms for Monitoring Wells

The mechanism(s) used for sampling should be specified in the QA Project Plan, including material composition of the device(s) and rationale for choosing the device(s).

- Choose a sampler to suit the most difficult parameter to be analyzed. For example, out-gassing and loss of volatile organics is a problem for most sampling mechanisms. The oxidation state of metals can change when exposed to atmospheric gases, leading to precipitation or dissolution not characteristic of the in-situ aquifer water.

Four characteristics that should be considered when selecting a sampling device are (from Barcelona, et al., 1985a):

1. The device should be simple to operate to minimize the possibility of operator error.
2. The device should be rugged, portable, cleanable, and repairable in the field.
3. The device should have good flow control to permit low flow rates (less than or equal to 100 mL/min) for sampling volatile chemical constituents, as well as high flow rates (greater than 1 L/min) for large-volume samples and for purging stored water from monitoring wells.
4. The mechanism should minimize the physical and chemical disturbance of ground water solution composition in order to avoid bias or imprecision in analytical results.

A laboratory assessment of some of the most commonly used sampling mechanisms provided the basis for evaluation in Table 5-6 (Barcelona, et al., 1984).

- Positive displacement bladder pumps meet all four of the suggested prerequisites above and are the most often recommended sampling device, especially for volatile or gas-sensitive constituents. Bladder pumps are available to fit inside most monitoring well casings (down to less than 2" I.D.).
- Dedicated bailers may be desirable for sampling to prevent contamination between wells if a bladder pump is not available. Sensitive parameters such as volatile organics and metals may be appreciably affected by sampling with a bailer if extreme care is not exercised. (NOTE: Even dedicated equipment [if not attached to the well] should be cleaned according to procedures similar to those

Table 5-6. Sampling mechanisms and performance ranking for monitoring wells
(Barcelona, et al., 1985a).

<u>Mechanism Category</u>	<u>Overall Performance Ranking</u>	<u>Remarks</u>
Positive displacement (bladder)	Above average	Expected to provide both efficient well purging and representative samples over a range of conditions with minimal difficulty in field operations.
Grab samplers (conventional bailer)	Average	Unsuitable for well purging; requires very careful operation and sample handling precautions under field conditions; field performance open to question.
(dual-check valve bailer)	Average	
(syringe pump)	Average to below average	
Positive displacement (mechanical)	Average to below average	Suitable for well purging; sampling performance very dependent on specific design and operational details.
Gas displacement (gas drive; not gas lift)	Average to	May be suitable for well purging if used in conventional installations; malfunctions are difficult to assess or repair; significantly lower recoveries of purgeable organic compounds and gases may occur depending on field conditions and operator experience.
Suction (peristaltic)	Below average	Suitable for well purging at depths to approximately 20 feet; significantly lower recoveries of purgeable organic compounds and gases will result from sampling with this mechanism.

recommendations under Section 5.f.1. after use. If any question exists regarding cleanliness of dedicated (or non-dedicated) equipment before sampling, equipment that will be exposed to sample water should again be cleaned).

- Sampling devices such as gas lift pumps that allow gases to contact sample water should not be used for collecting ground water samples.

5.g.2. Materials Used for Sampling Equipment

- Sampling equipment should be composed of materials that guarantee the most sensitive parameter(s) will not be affected by chemical decomposition of equipment, microbial colonization, sorption to and/or leaching from sampling materials.
- The relative surface contact of a water sample with sample tubing is six times greater than contact with the well casing (when sample collected from 1/4-inch tubing and 2-inch O.D. casing at a rate of 100 mL/min. (Barcelona, et al., 1985b). Therefore, choosing sample tubing components is especially important.
- If sampling for organics, teflon tubing is recommended, but polypropylene and linear polyethylene are acceptable. Silicone, neoprene, viton, and PVC should not be used for sampling delivery tubing, as these tend to sorb organics to a great extent, although all tubing sorbs dissolved organics to some degree (Barcelona, et al., 1985b).
- It is recommended if sample tubing is used, that tubing be changed or cleaned after purging and before sampling (especially for organics). If a bailer is used to purge, a clean bailer should be used to sample.
- In addition to tubing, no sampling equipment should be used that contains neoprene fittings or impellers. Also unacceptable are PVC bailers, tygon tubing, silicone rubber bladders, non-linear polyethylene vitron (EPA, 1985). Table 5-7 lists typical types of rigid materials used in monitoring devices and the advantages and disadvantages of each. Again, teflon is the highest rated material. Low-carbon, galvanized, and carbon steel are not recommended components for ground water sampling devices.

5.g.3. Sample Collection

Two references for guidance on parameter choice and sample-handling procedures for detection and assessment types of ground water monitoring, are Barcelona, et al. (1985a), Section 3 and Gibb (1983).

The following aspects of sample collection should be addressed in each GWMA QA Project Plan at a minimum. Deviations from or modifications of the recommendations should have a sound basis.

Table 5-7. Recommendations for rigid materials in sampling applications (in decreasing order of preference)
(Barcelona, et al., 1985a).

<u>Material</u>	<u>Recommendations</u>
Teflon* (flush threaded)	Recommended for most monitoring situations with detailed organic analytical needs, particularly for aggressive, organic leachate impacted hydrogeologic conditions. Virtually an ideal material for corrosive situations where inorganic contaminants are of interest.
Stainless Steel 316 (flush threaded)	Recommended for most monitoring situations with detailed organic analytical needs, particularly for aggressive, organic leachate impacted hydrogeologic conditions.
Stainless Steel 304 (flush threaded)	May be prone to slow pitting corrosion in contact with acidic high total dissolved solids aqueous solutions. Corrosion products limited mainly to Fe and possibly Cr and Ni.
PVC (flush threaded) other noncemented connections, only NSF** approved materials for well casing or potable water applications	Recommended for limited monitoring situations where inorganic contaminants are of interest and it is known that aggressive organic leachate mixtures will not be contacted. Cemented installations have caused documented interferences. The potential for interaction and interferences from PVC well casing in contact with aggressive aqueous organic mixtures is difficult to predict. PVC is not recommended for detailed organic analytical schemes.
Low-Carbon Steel; Galvanized Steel; Carbon Steel	Recommended for monitoring inorganic contaminants in corrosive, acidic inorganic situations. May release Sn or Sb compounds from the original heat stabilizers in the formulation after long exposures. May be superior to PVC for exposures to aggressive aqueous organic mixtures. These materials must be very carefully cleaned to remove oily manufacturing residues. Corrosion is likely in high dissolved solids acidic environments, particularly when sulfides are present. Products of corrosion are mainly Fe and Mn, except for galvanized steel which may release Zn and Cd. Weathered steel surfaces present very active adsorption sites for trace organic and inorganic chemical species.

*Teflon is a trademark of DuPont, Inc.

**National Sanitation Foundation approved materials carry the NSF logo, indicative of the product's certification based on meeting industry standards for performance and formulation purity.

- If sampling non-monitoring wells (e.g., public supply or private domestic wells), collect samples as close to the well head as possible. If a tap is the closest access, ensure that faucet does not have an aerator, especially if sampling for volatile organics.
- If down-hole sampler is used (e.g., for monitoring wells), wells should be sampled in order from lowest to highest contamination (e.g., upgradient wells first).
- Whether contamination levels are known or not, sampling device, tubing, and in-line, flow-through measuring system should be cleaned between wells as recommended for purging equipment, Section 5.f.1.
- At each well, the following order is recommended for collecting samples (as applicable):
 1. Volatiles
 2. TOC, TOX
 3. Constituents to be field filtered or field analyzed
 4. Large-volume samples for extractable organics, total metals, nutrients, and major cations and anions
- Figure 5-3 shows a well-designed scheme for ground water sample handling.
- Each QA Project Plan must include step-by-step details of sample collection procedures to be followed for each sampling event. These procedures must address the need for high-quality sampling information that is as representative of the in-situ ground water as possible. Any special procedures that are necessary for collecting or measuring particular sample parameters should also be described. The following areas should be included and elaborated upon. Changes or modifications of field procedures should be recorded on the Sample Alteration Checklist (Figure 5-2) and conveyed to the Lead Agency Project manager and Ecology Project Officer in a specified time period (e.g., two days, depending on the extent of the modification).
- In the same field notebook that purging information is recorded in (Figure 5-1), document sample collection information in format similar to Tables 5-3 and 5-4. Any deviation from stated procedures should be documented in field notes.
- One person in the sampling team should be responsible for documenting details of the following field procedures in the field notebook. The name of the recorder should be indicated on the report form (Figure 5-1).
- Before actual samples are collected, a sample of the final rinse water should be collected for critical parameters from the sampling device (e.g., pH, metals, and/or organics as applicable).

- If sampling for organics or low levels of inorganics, clean gloves made of appropriate material (e.g., surgical gloves) should be worn by everyone in contact with sampling equipment (especially if a down-hole sampler is used and/or samples are filtered in the field). Gloves also should be worn while equipment is being cleaned and handled after cleaning.
- The following suggestions should be followed as applicable to help ensure that representative samples are collected (U.S. EPA, 1985).
 - Positive displacement bladder pumps should be operated in a continuous manner so that they do not produce pulsating samples that are aerated in the return tube or upon discharge.
 - Check-valves should be designed and inspected to assure that fouling problems do not reduce delivery capabilities or result in aeration of the sample.
 - Sampling equipment (e.g., especially bailers) should never be dropped into the well because this will cause degassing of the water upon impact.
 - The bailer's contents should be transferred to a sample container in a way that will minimize agitation and aeration.
 - Clean sampling equipment should not be placed directly on the ground or other potentially contaminated surfaces prior to insertion into the well.
 - Bailer line must be dedicated so that it does not serve as a potential contaminant pathway. Monofilament line is recommended.
- Sample intake or bailer should be positioned at the same depth as the well screen for sample collection. Water samples should not be collected from the water column surface (New Jersey Dept. of Environmental Protection, 1983). (Immiscible layers [e.g., oil or gasoline] should, however, be sampled from the top of the water column.)
- In-line, flow-through bladder pump system is strongly recommended for sampling (see Garske and Schock, 1986; Barcelona, et al., 1985a; or NCASI, 1982 for details of setup and operation). Figure 5-4 shows a recommended flow-through system design.
- If an in-line, flow-through device is used for indicator parameter analyses, measurements should be recorded just before and immediately after sampling, as described under Purging, Section 5.f. (temperature, pH, specific conductance, and Eh). Samples should be collected after purging volume has been removed and purging parameters show relatively stable readings (e.g., when readings during two consecutive well volumes are within ± 10 percent).

SCHEMATIC OF SAMPLE HANDLING STEPS FOR GROUNDWATER

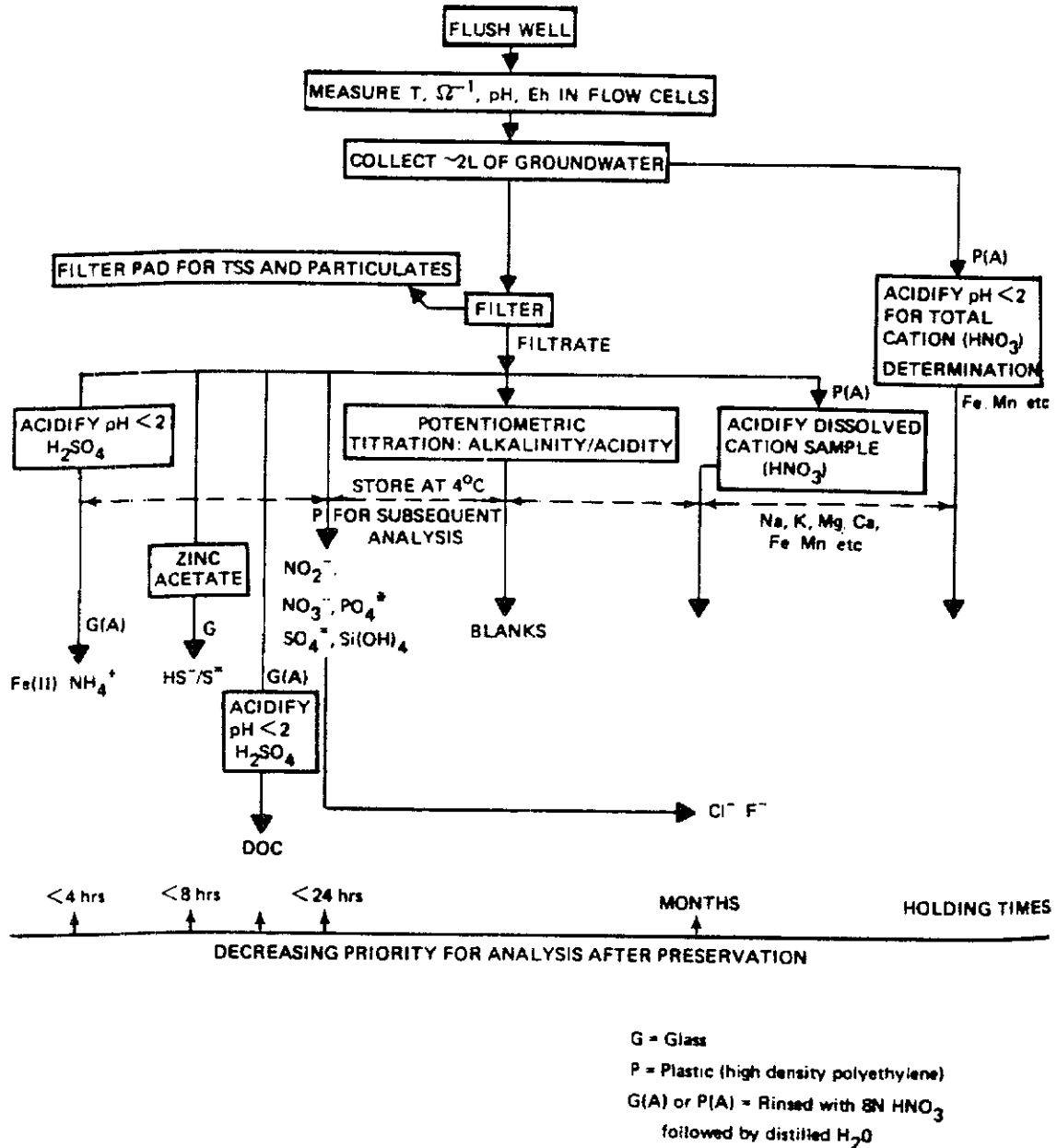


Figure 5-3. Schematic of sample handling steps for ground water (from Barcelona, et al., 1985).

- If in-line, flow-through sampling system used, sample should be collected from bypass or "tee" in tubing so that sample water does not pass electrodes or thermistors.
- Any turbulence introduced into the sample can affect metals, volatile organics, and many other chemical constituents. Turbulence, agitation, and exposure of sample water to the atmosphere should be minimized.
- Gibb (1985) suggests that bottom-draw bailers with dual check-valves are better than single check-valves for sampling volatiles and other sensitive constituents.
- Flow rate during sampling for volatile organics, metals, or other pH-, redox-, or gas-sensitive parameters should not exceed 100 mL/min. An exception can be made for large-volume samples that are not extremely sensitive.
- If sampling with a pump, sample discharge tube should be directed to the bottom of the sample bottle, especially for pH-, redox-, or gas-sensitive constituents. Samples for volatile organics, total organic halogens, or total organic carbon should also overflow a minimum of 1.5 volumes and be capped immediately. For volatile organics, after allowing a 40 mL vial to overflow at least 1.5 volumes, carefully cap tightly to avoid trapping any air. Invert vial and tap the side. If even a tiny bubble is visible, empty vial and sample again.
- If sampling from a well head, decrease discharge to a trickle, especially when collecting volatile organic samples. Allow vial to overflow with sample at least 1.5 volumes and carefully screw on teflon-lined silicon septum cap tightly to avoid trapping any air. Check volatile vials for air bubbles as described above.
- If sampling with a dual check valve bailer, collect sample from bottom check valve, slowly discharging water into sample container. Samples for volatiles, pH-, redox-, or gas-sensitive constituents should not be poured from top of a bailer. Check volatile vials for air bubbles as described above.
- If a bailer is used for sampling pH-, redox-, or gas-sensitive parameters, aeration is usually a problem. A study funded by the Electric Power Research Institute (1985) indicated that a more representative metals sample can be collected with a bailer if a clean tube made of an appropriate material is extended to the bottom of the bailer filled with sample. The bailer is then gently tipped to collect sample from the tubing without emptying the bailer completely. Another suggestion made by the authors of the study is to attach the filter to the tubing to avoid exposure to the atmosphere (if the sample requires filtering).
- Samples taken from a sampling device or from a well head should be collected directly into the sample bottle. Avoid transferring from one container to another.

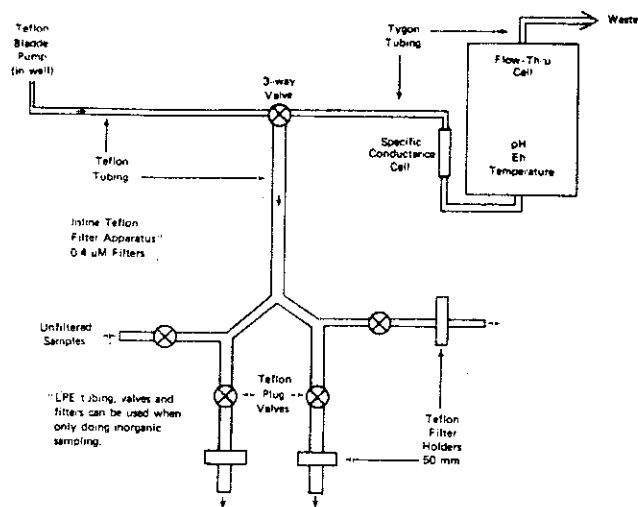


Figure 5-4. Diagram of valving and sampling system enabling in-line filtration of water samples, and sequential switching of membrane filters if they clog (from Garske & Schock, 1986).

- Do not field-filter organics samples.
- If an in-line, flow-through system is not used for measuring purging parameters, temperature, pH, and specific conductance should be measured and recorded after purging according to procedures outlined under the Calibration and Purging, Sections 5.c. and 5.f.
- If a pump is used, it should not be turned off until all samples have been collected.
- Do not place sampling probes or electrodes (or anything else except preservatives) in sample bottles to be used for laboratory analyses.
- If samples require preservation (according to the specified acceptable method), ensure that appropriate amount of preservative is added to appropriate containers. Or, if preservatives are added to containers before going into the field, check to make sure that containers have preservative inside before adding sample.
- For non-monitoring wells, ask owner or operator if there is any water treatment and if so, where in the distribution system. Sample water prior to any treatment (water softener, disinfection, etc.). If a sample is taken following some type of treatment or screen, record the size, type, and purpose of the unit in the field notebook.
- Labels must be attached to all sample containers. Labels should be filled out with waterproof ink. The QA Project Plan should specify the type of labels to be used and precautions to ensure that labels are not separated from samples and do not become illegible during transit. Information on the label should include:
 - sample number
 - constituent(s) to be analyzed
 - date and time sample collected
 - initials of collector
 - any remarks that may help laboratory analyst or others (e.g., color, odor, turbidity, etc.)

Sample location should not be specified on sample labels, but must be noted in field notebook. This way the laboratory analyst will not be biased when analyzing duplicates, blanks, and spike samples. (A portion of the blanks may be labeled as such so that the laboratory can tell immediately if a contamination problem exists.) See Section 5.h. for more information on quality control samples.

5.g.4. Sample Filtration

The following aspects of sample filtration in the field, if used in the project, should be addressed in the QA Project Plan, and specific details of standard protocols provided.

- If material is suspended in sample water after purging, field-filtering is recommended for dissolved metals, total dissolved solids, alkalinity/acidity, major ions, and other constituents that tend to react with solid materials.
- The recommended technique for field-filtering is as part of an in-line, flow-through system. This setup (shown in Figure 5-4) minimizes contamination, exposure to the atmosphere, and depressurization effects which can prevent collection of representative samples (Garske and Schock, 1986). Pump pressure is the driving force.
- If in-line, flow-through filtering is not used, sample water should be filtered immediately under nitrogen pressure through a clean 0.45 μ m glass fiber or teflon filter directly into the sample bottle. (The first 100 mL of filtrate should be discarded with each new filter.)
- Vacuum filtration should not be used for any ground water constituents.
- If sampling results conclusively show no significant difference between filtered and non-filtered samples, the project officer may waive the filtering requirement if it exists.
- Extreme care should be taken to avoid contamination during filtering. Forceps and other equipment that will contact the filters or other equipment that may contact the sample should be acid-washed (0.1 N HCl) and rinsed well with distilled or de-ionized water before use. Gloves should be worn, especially during all filtering procedures (and during the entire sampling process).
- Glass fiber filters also should be rinsed in dilute acid (0.1 N HCl) and then distilled water for sampling metals or nutrients.

5.h. Field Analyses

The following techniques and strategy for field analyses or those appropriate for the particular GWMA data collection project should be detailed in the QA Project Plan.

- Alkalinity values change very rapidly in weakly buffered water. Therefore, alkalinity samples (if collected) should be titrated in the field as soon as possible according to the specified accepted method (see Section 6). (If there is sufficient indication that there is no significant difference between alkalinity values measured immediately after sample collection and those measured within 24 hours of collection (U.S. EPA, 1979c maximum holding time), then the project officer

can determine that samples may be held up to 24 hours before analysis.)

- pH and temperature must be measured on-site immediately upon collection (see Section 5.c.) and during purging (see Section 5.f.).
- Specific conductance should also be measured on-site and during purging (see Section 5.f.).
- Field analyses for certain other inorganics (those that change rapidly despite preservation) are useful for comparison with or substitution for samples preserved and analyzed in the laboratory (e.g., ferrous iron, sulfide, nitrite, or ammonia) (Barcelona, et al., 1985a).
- Dissolved oxygen and Eh values must be measured in the field immediately upon collection (if the Winkler Method is used, dissolved oxygen must be fixed in the field, kept in the dark, and titrated within 4 to 8 hours). Exposure to the atmosphere should be minimized to obtain meaningful dissolved oxygen or Eh values.
- pH, temperature, Eh, and dissolved oxygen measurements made in the laboratory cannot be considered representative of the in-situ ground water.
- All equipment that comes in contact with sample water should be cleaned after each well is sampled (see Section 5.f. for cleaning procedures).

5.i. Field Quality Control Checks (blanks, duplicates, spike samples, and standards)

- The following are recommended levels for quality control check sampling for ground water sampling events (shown in Table 5-8). Modifications of the frequency of quality control check samples should be explained in the QA Project Plan or other written justification. Specifics of how quality control check samples are to be managed and handled should also be included in each QA Project Plan.
- At least one transport blank for each of the most important, and especially the most sensitive, parameters sampled each sampling day or one per 20 samples, whichever is greatest. The transport blank is a clean sample container from the same batch of sample containers used for collecting real samples that day filled with laboratory distilled, distilled-deionized, or organic-free water. The transport blank is carried into the field and treated the same as a normal sample although it is never opened. It is handled, stored, and submitted to the lab in the same way as the other samples for which it serves as a blank. The transport blank should not be labeled as such.
- At least one field blank should be prepared in the field every sampling day for each of the most important and sensitive

Table 5-8. Field quality control check samples.

<u>Characteristic of Interest</u>	<u>Sample Type</u>	<u>Recommended Frequency for Most Important or Sensitive Parameters</u>
Field/transport contamination	Transport blank	One per 20 samples; at least one per sample day, if less than 20 samples. (This is especially important for organics.)
Field/transfer contamination	Field blank	One per 20 samples; at least one per sample day, if less than 20 samples.
Field/filtrating contamination	Field filtration blank	One per 20 samples; at least one per sample day if less than 20 samples.
Sampling equipment contamination	Equipment blank	One per well sampled unless previous data collected by same samplers indicate that one per sample day is sufficient.
Precision (field)	Field duplicates	Organics: one per 20 samples. Inorganics: one per 10 samples. (At least one for each sampling day for both organics and inorganics if fewer samples collected. For volatiles, one set of triplicates should be collected.)
Accuracy	Standard samples	One per 20 samples; at least one per sampling.
	Field split spike samples	One per 20 samples; at least one per sampling.
	Field split samples	One per quarter (or biannum) for the major parameters of concern sent to outside laboratories.
Container contamination	Empty container	One per container lot.

constituents, if not each constituent, or one per 20 samples, whichever is greater. A field blank is prepared by adding laboratory water (distilled, distilled-deionized, or organic-free water) to a sample bottle. The field blank should be preserved, handled, stored, and transferred to the laboratory the same way a real sample would be. Field blanks should be labeled so that the laboratory analyst does not know that the samples are different from real samples.

- A field filtration blank should be collected for constituents filtered in the field. A clean container is filled with de-ionized, distilled water; carried into the field; and filtered in the same manner as real samples. The filtered water is transferred into another clean container and treated as a normal sample. At least one field filtration blank should be collected per sampling day that field filtering is carried out.
- If a ground water sampling device is used, one equipment blank should be collected for each of the most important and sensitive parameters sampled. After the sampling device is cleaned and before the sample is collected, fill the device with final rinse water (distilled, distilled-deionized, or organic-free water) and pump or drain sample from device. For the first few sampling trips it is recommended that equipment blanks be collected before each well is sampled. If contamination between wells does not appear to be a problem and the same crew does the sampling, fewer equipment blanks may be necessary in the future. The Lead Agency Project Manager should be consulted in determining the appropriate number of equipment blanks after initial results are reviewed. The Ecology Project Officer should also be kept informed regarding changes in equipment blank procedures.
- A duplicate sample for the major inorganic parameters should be collected for every 10 wells sampled, and an organics duplicate for every 20 wells sampled (at least one triplicate for volatiles) or a minimum of one duplicate for each major parameter if fewer than 10 (for inorganics) or 20 (for organics) samples collected per sampling day. Duplicate or triplicate samples should be collected immediately after the real sample using the same procedure. Duplicates and triplicates should be labeled so that the laboratory analyst does not know that they are duplicates.
- Occasional split field samples should be sent to one or more outside laboratory(ies) as a quality control check (e.g., one per quarter for the major parameters of interest).
- At least one standard sample should be prepared in the field for every 10 samples, or at least one per sampling day if less than 10 samples are collected. Standard solutions should be prepared in the laboratory and brought into the field. A sample bottle is then carefully filled with the standard solution (traceable to a known standard) and preservative added. Or the proper solvent is added to the sample container and this material is spiked with stock solution. EPA Performance Evaluation samples or standard reference

Table 5-9. Field standards and sampling spiking solutions (from Barcelona, et al., 1985a).

Sample Type	Volume	Composition	Field standard (concentration)	Stock Solution For Field Spike of Split Samples		
				Solvent	Concentration of Components	Field Spike Volume
Alkalinity	50 mL	Na ⁺ , HCO ₃ ⁻	10.0; 25 (ppm)	H ₂ O	10,000; 25,000 (ppm)	(50 µL)
Anions	1 L	K ⁺ , Na ⁺ , Cl ⁻ , SO ₄ ⁻⁻	25, 50 (ppm)	H ₂ O	25,000; 50,000 (ppm)	(1 mL)
Cations	1 L	Na ⁺ , K ⁺ Ca ⁺⁺ , Mg ⁺⁺ , Cl ⁻ , NO ₃ ⁻	5.0; 10.0 (ppm)	H ₂ O, H ⁺ (acid)	5,000; 10,000 (ppm)	(1 mL)
Trace metals	1 L	Cd ⁺⁺ , Cu ⁺⁺ , Pb ⁺⁺ Cr ⁺⁺⁺ , Ni ⁺⁺ , AG Fe ⁺⁺ , Mn	10.0; 25.0 (ppm)	H ₂ O, H ⁺ (acid)	10,000; 25,000 (ppm)	(1 mL)
TOC	40 mL	Acetone KHP	0.2; 0.5 (ppm-C) 1.8; 4.5 (ppm-C)	H ₂ O	200; 500 (ppm-C) 1,800; 4,500 (ppm-C)	(40 µL)
TOX	500 mL	Chloroform 2,4,6 Trichlorophenol	12.5; 25 (ppb) 12.5; 25 (ppb)	H ₂ O/poly* (ethylene glycol)	12.5; 25.0 (ppm) 12.5 25.0 (ppm)	(500 µL)
Volatiles	40 mL	Dichlorobutane, Toluene Dibromopropane, Xylene	25; 50 (ppb)	H ₂ O/poly* (ethylene glycol)	12.5 25.0 (ppm)	(40 µL)
Extractables A	1 L	Phenol Standards	25; 50 (ppb)	Methanol**	25; 50 (ppm)	(1 mL)
Extractables B	1 L	Polynuclear Aromatic Standards	25; 50 (ppb)	Methanol	25; 50 (ppm)	(1 mL)
Extractables C	1L	Standards as Required	25; 50 (ppb)	Methanol	25; 50 (ppm)	(1 mL)

* = 75:25 Water/Polyethylene Glycol (400 amu) Mixture

** = Glass Distilled Methanol

materials also can be used. Standard samples should also be labeled so that it is not obvious to the laboratory analyst that they are standards. Table 5-9 shows an example of standard solutions, although the concentrations are quite a bit higher than would be expected in uncontaminated ground waters. Therefore, lower level standards for most constituents are recommended. [Methylene chloride should not be used as a standard (Barcelona, et al., 1985a).]

- It is recommended that split spike samples be collected every 10 samples for sensitive or important constituents, or one per sampling day if less than 10 samples collected. Split spike samples can be prepared in two ways.
- One way is to collect a double volume of sample in a clean container and carefully transfer into two clean sample containers. Avoid any unnecessary sample agitation or manipulation as this greatly increases the potential for contamination and alteration of chemical constituents. The spike solution can then be added (extremely carefully) in the field. This avoids any inadvertent bias or special handling by the laboratory. The risk of contamination, however, is quite high if extreme caution is not used when spiking in the field.
- The alternative is to submit the split samples to the laboratory and the spike can be added there. This type of laboratory spike is recommended in addition to the field spike (if field spiking is performed). The possibility of contamination is less for a lab spike, although total analytical non-bias is forfeited.
- Spikes should be added to fortify the sample by 30 to 500 percent of expected concentrations. The QA Project Plan should describe the strategy and mechanics for sample spiking in the field. Specifics of spiking should be included in field notes, and submitted with sample results, including the standards used, concentration of spike material, and volume of the spike.
- One empty container per container batch for each of the most important or sensitive parameters of interest should be taken into the field to test for container contamination. The container should not be opened, but submitted to the laboratory labeled with a sample number as the other samples.

5.j. Sample Storage, Packaging, and Transport

- Specific procedures and timeframes should be specified in each QA Project Plan for sample storage, packaging, and transport. The following should be included:
 - Procedure for notifying the laboratory of the sampling schedule and estimated time of sample arrival. Arrangements for sample transport and delivery to the laboratory should be verified in advance

of sampling to ensure that holding time requirements are met. Precautions should be planned to prevent sample loss, damage, or mishandling en route to the laboratory.

- Samples that require cooling should be put on ice immediately following sample collection.
- Organic samples should not be stored in styrofoam or in containers that have previously held organic solvent.
- Every attempt should be made to prevent organic reagent vapors (e.g., cleaning solvents) from getting anywhere near organic samples which are easily contaminated.
- Sample packaging should fulfill the requirements of EPA's "User's Guide to the Contract Laboratory Program." Breakable containers should be packaged to prevent leaking and breakage. Padding and absorbent material must not be capable of reacting with sample contents.
- Any unique situations related to sample storage or transport should be recorded in the field notebook (e.g., delay in cooling, very warm or cold day, etc.).
- Procedures should be documented and implemented to assure that maximum holding times (as shown in Section 6) are observed. Actual holding periods must be recorded for each sample and included in data reports.

6. ANALYTICAL SYSTEM (LABORATORY)

Any laboratory that carries out physical, chemical, or biological analyses on samples as part of a Ground Water Management Areas Program (Chapter 173-100 WAC) must have a written in-house Quality Assurance/Quality Control (QA/QC) Procedures Manual that specifies procedures for laboratory operation and analytical protocols for constituents analyzed. This QA/QC system must be acceptable to the Ecology Project Officer. The laboratory's QA/QC procedures will be compared to those required or recommended under major EPA programs (U.S. EPA, 1979a, 1979b, 1982, 1984; EPA Contract Laboratory Program procedures) in order to evaluate adequacy. Some areas of particular interest that should be covered as a minimum in the laboratory's QA/QC plan are described below. If the laboratory's QA/QC plan does not cover these areas or is less stringent in its requirements, the contract should specify that the applicable recommendations below be carried out as part of each analysis. Additional internal QA/QC procedures are encouraged.

Laboratories should be required to submit evidence of their routine accuracy and precision for analyses to be carried out on a project before the choice of laboratories is made. It is also recommended that laboratories analyze a blind standard reference sample or split field samples to compare actual results before a laboratory is approved for the GWMA sample analysis work.

6.a. Minimum Qualifications for Laboratory Analysts

- Minimum qualifications for analysts carrying out specific types of analyses on GWMA samples are shown in Table 6-1.

As part of this documentation, the analytical laboratory will submit and update information to demonstrate that personnel carrying out analyses on metals and organics samples meet the following minimum requirements (from U.S. EPA Contract Laboratory Program Statement of Work for Metals Analysis and Statement of Work for Organics Analysis). Experience is defined as more than 50 percent of the person's productive work time in active participation of the specified task.

Metals

- The Flameless Atomic Absorption (AA) Spectroscopist responsible for the work on this contract must have at least one (1) year's experience in the operation of flameless AAS on environmental samples.
- The Flame and Cold Vapor Atomic Absorption Spectroscopist responsible for the work on this contract must have at least nine (9) months' experience in the operation of flame and cold vapor AA on environmental samples.
- The Inorganic Sample Preparation Expert performing sample preparation for the work on this contract must have at least three (3) months' experience in the preparation of AA samples and standards.

Table 6-1. Skill-time rating of standard analytical operations
(from EPA, 1979).

Measurement	Skill Required (Rating No.) ¹
Simple Instrumental:	
pH	1
Conductivity	1
Turbidity	1
Color	1
Dissolved Oxygen (Probe)	1,2
Fluoride (Probe)	1,2
Simple Volumetric:	
Alkalinity (Potentiometric)	1
Acidity (Potentiometric)	1
Chloride	1
Hardness	1
Dissolved Oxygen (Winkler)	1,2
Simple Gravimetric:	
Solids, Suspended	1,2
Solids, Dissolved	1,2
Solids, Total	1,2
Solids, Volatile	1,2
Simple Colorimetric:	
Nitrite N (Manual)	2
Nitrate N (Manual)	2
Sulfate (Turbidimetric)	2
Silica	2
Arsenic	2,3
Complex, Volumetric, or Colorimetric:	
BOD	2,3
COD	2,3
TKN	2,3
Ammonia	2,3
Phosphorus, Total	2,3
Phenol (Distillation Included)	2,3
Oil and Grease	2,3
Fluoride (Distillation Included)	2,3
Cyanide	2,3
Special Instrumental:	
TOC	2,3
Metals (by AA), No Preliminary Treatment	2,3
Metals (by AA), With Preliminary Treatment	2,3
Organics (by GC), Pesticides, Without Cleanup	3,4
Organics (by GC), Pesticides, With Cleanup	3,4

¹ Skill-required rating numbers are defined as follows:

- 1—aide who is a semiskilled subprofessional with minimum background or training, comparable to GS-3 through GS-5.
- 2—aide with special training or professional with minimum training with background in general laboratory techniques and some knowledge of chemistry, comparable to GS-5 through GS-7.
- 3—experienced analyst capable of following complex procedures with good background in analytical techniques, professional, comparable to GS-9 through GS-12.
- 4—experienced analyst specialized in highly complex procedures, professional, comparable to GS-11 through GS-13.

² Rate depends on type of samples.

Organics

- The contractor laboratory's GC/MS operators performing work on this contract shall have at least nine (9) months' experience in the operation of the GC/MS on environmental samples.
- The contractor laboratory's mass spectral interpretation specialist performing work on this contract shall have at least two (2) years' experience in the interpretation of mass spectra gathered in GC/MS analysis.
- The contractor laboratory's extraction and concentration specialist performing work on this contract shall have at least one (1) year's experience in the preparation of extracts from environmental samples.
- The contractor laboratory's purge and trap specialist performing work on this contract shall have at least six (6) months' experience using the purge and trap technique for volatile organics.
- The contractor laboratory's pesticide residue analysis specialist performing work on this contract shall have at least two (2) years' experience in organochlorine pesticide residue and PCB analysis, including cleanup procedures such as column chromatography, on environmental samples.

The contractor laboratory shall preserve all sample extracts after analysis in bottles/vials with teflon-lined septa and maintained at 4 degrees C. Within 180 days after data submission, the laboratory QA officer shall request the Lead Agency Project Manager for authorization to dispose of sample extracts or provide the sample extracts to Ecology. Extract disposition shall comply with the Lead Agency Project Manager's determination after consulting with the Ecology Project Officer.

Changes in personnel during the course of the project should be reported immediately to the Lead Agency Project Manager and Ecology Project Officer.

6.b. Calibration Procedures and Preventive Maintenance for Laboratory Instruments

Each Ground Water Management Area QA Project Plan should include a description of calibration procedures, frequency, and standards used to ensure that the laboratory equipment functions properly. Calibration and preventive maintenance data should be recorded in instrument log books at established intervals. Acceptability requirements for equipment calibration should be established. If calibration standards are used, they should be traceable to primary standards that are available (e.g., National Bureau of Standards).

Table 6-2. Calibration procedures and frequency (adapted from Tetra Tech, Inc., 1986).

<u>Variable/System</u>	<u>Calibration Frequency</u>	<u>Standard</u>	<u>Manufacturer and Model Number</u>	<u>Preventive Maintenance</u>
Analytical laboratory: mass spectrometer with capillary column gas chromatograph	Beginning of each shift, every 12 hours or every 10 - 12 runs, whichever is more frequent, and end of each shift.	DFTPP, BFB, and response standards		
Electron capture detector with gas chromatograph	Beginning of each shift, every 6 hours or every 12 hours if > hour runs, and end of each shift.	Analyte standards		
Atomic absorption or inductively coupled plasma spectrometer	Every 2 hours or ever 10 samples, whichever is more frequent.	Analyte standards		
alpha spectrophotometer	Daily	alpha source		
Elemental analyzer (carbon)	Standard curve (minimum of 4 points) at start of run and one standard every 10 samples thereafter.	Acetanilide or other carbon source		
Analytical balances	Quarterly over complete range. Annually by qualified balance analyst.	Class S weights		Users should be encouraged to protect and care for balance. Instructions for use fixed beside balance.
Distilled water still	N/A			
Water de-ionizer				
Activated charcoal system for organic-free water				Change charcoal filter per manufacturer's recommendations.

A table showing specific equipment used, summary of calibration procedures, frequency, and preventive maintenance procedures for each analysis should also be provided. An example is shown in Table 6-2.

6.b.1. Calibration for Inorganics

Follow calibration procedures required in the accepted method (listed under 6.c. or others in Table 1B of U.S. Federal Register, October 26, 1984, 40 CFR Part 136, with the following additions or emphasis.

- Materials and reagents used for preparation of primary standard stock solutions should be documented in the QA Project Plan. Likewise, procedures for preparing primary, secondary, and, if applicable, tertiary standards should be included in the QA Project Plan, along with frequency of preparation.
- Calibration curves should be prepared daily and with each setup from analysis of a minimum of four standards (one near but above the method detection limit, and the other three to span the expected range of samples), and one blank (distilled, distilled-deionized, or organic-free water used in standards preparation).
- A calibration curve should be developed for every hour of continuous sample analysis.
- Calibration curves should be submitted with sample results to the Lead Agency Project Manager and Ecology Project Officer.
- Laboratories should submit copies of control charts for relevant analyses at least quarterly to the Contractor Project Manager. These copies should also accompany data submitted to the Lead Agency and Ecology (U.S. EPA, 1979b).

6.b.2. Calibration of the gas chromatograph for organics

Follow calibration procedures required in the accepted method (listed under 6.c.) that the Ecology Project Officer approves with the following additions.

- Calibration curves should be prepared from analysis of a minimum of four standards (one near but above the method detection limit) and one blank (organic-free water).
- Calibration should be done at the beginning and end of each analysis session. Continuing calibration verification should be done by analyzing an outside standard (from EPA, National Bureau of Standards, or standards traceable to one of these) after every ten samples or every two hours during the analysis run, whichever is more frequent. The outside standard should be near the mid range of the calibration curve. Results for standard reference samples of metals should not be outside the range of 85 to 115 percent of the true value (mercury may be 80 to 120 percent). The instrument

must be recalibrated if standard results are outside these limits and the ten samples analyzed since previous calibration reanalyzed (U.S. EPA Contract Laboratory Program Statement of Work for Metals).

- Calibration curves should be submitted to the Ecology Project Officer as part of the sample results package.

6.c. Analytical Methods

- Methods approved or recommended by the U.S. EPA [Federal Register, October 26, 1984, 40 CFR Part 136 or EPA (1982)] should be used to analyze all Ground Water Management Areas water samples, along with corresponding quality assurance procedures. If more than one method is approved by EPA for a particular type of analysis, it is recommended that the method with the lowest method detection limit be used (especially for trace metals and organics). It also is recommended that analysts strive for detection limits below the method detection limits, especially if samples are for background water quality characterization.
- Table 6-3 lists the U.S. EPA methods, detection limits, and holding times for some of the constituents commonly analyzed in ground water. (Extraction methods for organics are not listed in Table 6-3, but are described in U.S. EPA (1982) and U.S. Federal Register, October 26, 1984, 40 CFR Part 136.
- If no adequate procedure is available from EPA, a written description of another procedure or reference to the procedure must be submitted to the Lead Agency Project Manager and Ecology Project Officer for approval before the procedure is used.
- If an inorganic sample value is outside the calibration range, the sample should be diluted once toward the mid-range of the calibration curve. (Organics should not be diluted)
- Any short- or long-term modifications made to approved methods should be conveyed to the Ecology project officer via the Sample Alteration Checklist (Figure 5-2).
- Each QA Project Plan will include a table showing the parameters to be sampled, the analytical method to be used for each, and the proposed detection limits.

6.d. Laboratory Quality Control checks (blanks, duplicates, spike samples, and standards)

Each in-house laboratory quality assurance program should include provisions for ensuring the proper purity of reagents, water used in cleaning and analyses, glassware, solvents, gases, etc. for particular analyses to be carried out on Ground Water Management Areas samples (see Chapters 4 and 5 of U.S. EPA [1979a] for minimum levels of effort necessary in this

area). These checks should be described or referred to in the QA Project Plan for each Ground Water Management Area sampling project.

Each in-house laboratory quality assurance program should include provisions for routinely assessing analytical accuracy and precision for each analysis. Chapters 5 and 6 of U.S. EPA (1979a) should be a guide.

If organic compounds are to be analyzed by the laboratory, the analyst must first (before analyzing any Ground Water Management Area samples) demonstrate one time that data of sufficient specified accuracy and precision can be produced. The steps listed in U.S. Federal Register October 26, 1984, 40 CFR Part 136 for each analysis under Item #8. "Quality Control" for initial method demonstration should be required for each analysis requested (even if the particular analysis and control limits are not specified in 40 CFR part 136).

Before any metals samples are analyzed for a Ground water Management Areas, the instrument detection limits must be determined (ug/L) and repeated at least quarterly while Lead Agency samples are being sent to the lab for analysis. The instrument detection limits (in ug/L) are calculated by multiplying by 3 the standard deviation obtained on three non-consecutive days from analysis of a standard metals solution (each analyte in reagent water) at a concentration of 3 to 5 times the usual instrument detection limit with seven consecutive measurements per day (EPA Contract Laboratory Program Statement of Work for Metals Analysis).

The QA Project Plan will specify the frequency of laboratory quality control check samples for metals, nutrients, organics, cations, and anions. The QC check samples recommended for these analyses are listed in Table 6-4. Definitions of the different check samples are provided below. Written justification for frequencies of check samples should be provided.

- One reagent blank for each reagent or solvent used should be analyzed periodically (not necessarily with each batch). The reagent blanks are prepared by treating each reagent as a sample and carrying a subsample of it through the whole analysis (EPA, 1979a).
- One method blank per sample batch. The method blank is prepared by carrying out the procedure as prescribed in the method, adding all reagents and solvents in the specified quantity. This is necessary to determine the cumulative interferences from reagents (EPA, 1979a). The method blank must be analyzed routinely during analysis of each batch of sample at the frequency specified by the method. (The method blank described here is the same as the reagent blank described in U.S. Federal Register [1984].) The method blank can use distilled, de-ionized, or organic-free water or solvents used in the analysis as part of the blank depending on the analysis.
- A calibration blank is necessary for metals analyses. This is usually prepared by diluting 2 mL of (1+1) HNO_3 and 10 mL of (1+1) HCl to 100 mL with de-ionized, distilled water. A sufficient amount should be prepared to flush the system between standards and samples (EPA Contract Laboratory Program Statement of Work for Metals Analysis).

Table 6-3. EPA Methods and Method Detection Limits for Typical Ground Water Analytes
(See Table 1B in U.S. Federal Register (October 24, 1984) 40 CFR Part 136
for additional approved methods for inorganics)

Parameter	Method Number EPA - Methods for Chemical Analysis of Wastes (EPA-600/4-79-020)	Method Detection Limit according to EPA-600/4-79-020 (ug/L)	EPA (1982) SW-846 Method Number	Method Detection Limit EPA (1982) SW-846 (ug/L) a/	Federal Register Part 136 Method Detection Limit (ug/L)	Maximum Holding Time (assuming proper preserva- tion) b/
<u>Metals and Major Ions</u>						
Aluminum	202.1, 202.2	3.0	-	-		6 mo.
Arsenic	206.2, 206.3	1.0, 2.0	7060	1.0		6 mo.
Barium	208.1, 208.2	100., 2.0	7080, 7081	100., 2.0		6 mo.
Cadmium	213.1, 213.2	5.0, 0.1	7130, 7131	5.0, 0.1		6 mo.
Calcium	215.1	10.0	-	-		7 days
Chromium	218.1, 218.2	50., 1.0	7190, 7191	50., 1.0		6 mo.
Chromium, Hexavalent	218.4		7196, 7197	Reverification		24 hrs
Copper	220.1, 22.2	20., 1.0	7210, 7211			6 mo.
Fluoride	340.1, 340.2, 340.3	100., 100., 50.	-	-		7 days
Iron	236.1, 236.2	30., 1.0	-	-		6 mo.
Lead	239.1, 239.2	100., 1.0	7420, 7421	100., 1.0		6 mo.
Magnesium	242.1	1.0	-	-		6 mo.
Manganese	243.1, 243.2	10., 0.2	-	-		6 mo.
Mercury	245.1, 245.2	0.2	7470	0.2		13 days
Nickel	249.1, 249.2	40., 1.0	7520, 7521	40., 1.0		6 mo.
Potassium	258.1	10.0	-	-		6 mo.
Selenium	270.2, 270.3	2.0	7740	2.0		7 days
Sodium	273.1	2.0	-	-		6 mo.
Zinc	289.1, 289.2	5.0, .05	7950, 7951			6 mo.

AV/WQ3/86/120201(1)

Table 6-3. EPA Methods and Method Detection Limits for Typical Ground Water Analytes (Continued)
(See Table 1B in U.S. Federal Register (October 24, 1984) 40 CFR Part 136
for additional approved methods for inorganics)

Parameter	Method Number EPA - Methods for Chemical Analysis of Wastes (EPA-600/4-79-020)	Method Detection Limit according to EPA-600/4-79-020 (ug/L)	EPA (1982) SW-846 Method Number	Method Detection Limit EPA (1982) SW-846 (ug/L) a/	Federal Register 40 CFR Part 136 Method Detection Limit (ug/L)	Maximum Holding Time (assuming proper preserva- tion) b/
<u>Conventionals</u>						
Alkalinity	310.1, 310.2					24 hrs.
Total dissolved solids	160.1					7 days
Ammonia	350.1, 350.2, 350.3	10., 50., 30.				24 hrs.
Nitrite	354.1	10.0				48 hrs.
Nitrate-Nitrite	353.1, 353.2, 353.3	10., 50., 10.0				24 hrs.
Chloride						
Sulfate	375.1, 375.2, 375.4	10,000, 500, 1,000				7 days
Sulfide	376.1, 376.2	1,000, 1,000				24 hrs.
Carbonate						
Bicarbonate						
Cyanide	335.1, 335.2, 335.3	1,000, 1,000, 5.0	9010			24 hrs.
Hardness	130.1, 130.2	10,000, 25,000				6 mo.
<u>Organics</u>						
Purgeable Halocarbons	(Halogenated Volatile Organics)		8010	0.03-0.5*	601	7 days/ 14 days
Total Organic Halides			8015	-	-	
Purgeable Aromatics (Aromatic Volatile Organics)	(Purge and Trap/ GC/MS)		8020	0.2-0.4*	602	7 days/ 14 days
Phenols			8040	0.1-16.0*	604	7 days/ 40 days

AV/WQ3/86/120201(2)

Table 6-3. EPA Methods and Method Detection Limits for Typical Ground Water Analytes (Continued)
(See Table 1B in U.S. Federal Register (October 24, 1984) 40 CFR Part 136
for additional approved methods for inorganics)

Parameter	Method Number EPA - Methods for Chemical Analysis of Wastes (EPA-600/4-79-020)	Method Detection Limit according to EPA-600/4-79-020 (ug/L)	EPA (1982) SW-846 Method Number	Method Detection Limit EPA (1982) SW-846 (ug/L) a/	Federal Register Part 136 Method Detection Limit (ug/L)	Federal Register Part 136 Method Detection Limit (ug/L)	Maximum Holding Time (assuming proper preserva- tion) b/
Phthalate Esters			8060	0.29-3.0*	606	0.29-3.0*	7 days/40 days
Organochlorine Pesticides and PCBs			8080	0.004-.176*	608	0.003-0.24*	7 days/40 days
Nitroaromatics and Isophrone			8090	0.06-5.0*	609	0.01-15.7	7 days/40 days
Organophosphorus Pesticides			8140	0.1-5.0*	-	-	7 days/40 days
Chlorinated Herbicides			8150	0.1-200*	-	-	7 days/40 days
Polynuclear Aromatic Hydrocarbons			8310	0.013-2.3*	610	0.013-2.3*	7 days/40 days
Chlorinated Hydrocarbons			8120	0.03-1.19*	612	0.03-1.19*	7 days/40 days
Purgables (Volatile Organics - GC/MS)			8240	nd-7.2*	624	nd-7.2*	7 days/14 days
Base Neutrals and Acids (GC/MS Method for semi- volatile organics: packed column technique)			8250	0.9-42*	625	0.9-42*	7 days/40 days
<u>Bacteria</u>							

Sample preparation and extraction methods as recommended in methods above.
(nd - not determined).

* - Depends on individual compound.

a/ U.S. Federal Register October 24, 1984. 40 CFR Part 136. Guidelines Establishing Test Procedures for the Analysis of Pollutants under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule.

b/ Where two times are given, the first is maximum time to extraction; the second is maximum time to instrument analysis. Samples should be analyzed as soon as possible after collection. Times listed are maximum times that samples can be held and results would still be considered valid.

AV/WQ3/86/L20201(3)

Table 6-4. Laboratory quality control check samples.

<u>Characteristic of Interest</u>	<u>Sample Type</u>	<u>Recommended Frequency</u>
Laboratory contamination	Reagent blank	When new reagent(s) used.
Laboratory contamination	Method blank	One per sample batch - analyzed every 10 samples at frequency specified in individual methods.
Instrument contamination (for metals)	Calibration blank	Use to flush system between standards and samples.
Laboratory contamination	Extraction blank	For organics, two per extraction batch.
Precision (laboratory)	Laboratory duplicates	One per ten samples (at least one per sample batch if less than 10).
Accuracy	EPA performance evaluation samples or standard reference materials	One per ten samples (at least one per sample batch if less than 10).
	Spike additions	One per ten samples (at least one per sample batch if less than 10).

- One duplicate sample should be prepared in the laboratory for every 10 project samples submitted (excluding those labeled as splits or blanks) or one per sample batch if less than 10 samples submitted.
- One EPA performance evaluation sample or standard reference material in the concentration range of samples being analyzed should be run according to instructions and method requirements. The standard reference material sample is to be analyzed once every 10 samples submitted.
- One out of every 10 samples submitted will be split and one half spiked (excluding those labeled as field splits or blanks). The concentration of the spike should be in the range of one to three times the expected sample concentration before spiking.

Metals Analysis

If the Furnace Atomic Absorption (AA) technique is used for metals analysis, the following QC analysis is recommended (from EPA Contract Laboratory Program Contract Statement of Work for Metals Analysis).

"1. All furnace analyses, except during Full Methods of Standard Addition (MSA), will require duplicate injections for which the average absorbance or "concentration" will be reported. All analyses must fall within the calibration range. The raw data package must contain both absorbance or "concentration" values, the average value, and the relative standard deviation (RSD) or coefficient of variation (CV). For concentrations greater than the method detection limit, the duplicate injection readings must agree within 20 percent RSD or CV, or the sample must be rerun once. If the readings are still out, flag the value with an "M" on the inorganics data sheet Form I.

2. All furnace analyses for each sample will require at least a single analytical spike to determine if the MSA will be required for quantitation. Analytical spikes are not required on the predigest spike samples. The spike¹ will be required to be at a concentration (in the sample) twice the required method detection limit. The percent recovery (%R) of the spike, calculated by the formula given in 8.b., will then determine how the sample will be quantitated as follows:

- a) If the spike recovery is less than 40 percent, the sample must be diluted and rerun with another spike. Dilute the sample by a factor of five to ten and rerun. This step must only be performed once. If after the dilution the spike recovery is still less than 40 percent, report data and flag with an "E" to indicate interference problems.

¹ Spikes are post-digest spikes to be prepared prior to analysis by adding a known quantity of the analyte to an aliquot of the digested sample. The unspiked sample aliquot must be compensated for any volume change in the spike samples by addition of deionized water to the unspiked sample aliquot.

- b) If the spike recovery is greater than 40 percent and the sample absorbance or concentration is less than 50 percent of the spike^{**}, report the sample as less than the method detection limit or less than the method detection limit times the dilution factor if the sample was diluted.
 - c) If the sample absorbance or concentration is greater than 50 percent of the spike² and the spike recovery is between 85 percent and 115 percent, the sample should be quantitated directly from the calibration curve.
 - d) If the sample absorbance or concentration is greater than 50 percent of the spike² and the spike recovery is less than 85 percent or greater than 115 percent, the sample must be quantitated by MSA.
3. The following procedures will be incorporated into MSA analyses.
- a) Data from MSA calculations must be within the linear range as determined by the calibration curve generated at the beginning of the analytical run.
 - b) The sample and three spikes must be analyzed consecutively for MSA quantitation (the "initial" spike run data are specifically excluded from use in the MSA quantitation). Only single injections are required for MSA quantitation.
 - c) Spikes¹ should be prepared such that:
 - Spike 1 is approximately 50 percent of the sample absorbance.
 - Spike 2 is approximately 100 percent of the sample absorbance.
 - Spike 3 is approximately 150 percent of the sample absorbance.
 - d) The data for each MSA analysis should be clearly identified in the raw data documentation along with the slope, intercept, and correlation coefficient (r) for the least squares fit of the data and the results reported on Form VIII. Reported values obtained by MSA are flagged on the data sheet (Form I) with the letter "s."
 - e) If the correlation coefficient (r) for a particular analysis is less than 0.995, the MSA analyses must be repeated once. If the correlation coefficient is still less than 0.995, the results on Form I must be flagged with "+."

¹ Spikes are post-digest spikes to be prepared prior to analysis by adding a known quantity of the analyte to an aliquot of the digested sample. The unspiked sample aliquot must be compensated for any volume change in the spike samples by addition of deionized water to the unspiked sample aliquot.

² "Spike" is defined as (absorbance or concentration of spike sample) minus (absorbance or concentration of the sample).

Organics Analysis

For trace organics analysis, the EPA Contract Laboratory Program Statement of Work for Organics Analysis should be followed in terms of sample preparation, storage, GC/MS calibration techniques, analytical techniques, QA/QC requirements, data analysis, and documentation (Forms I - X, as applicable in Appendix).

- All records of QC check samples must be documented and included with sample results for the associated sample batch.
- Procedures must be specified in the QA Project Plan for responding to situations where QC checks indicate a problem or do not meet acceptance criteria for the method. (For metals, the acceptance range for spike recoveries should be within 80 to 120 percent. Acceptance criteria for metals blanks should be the specified required detection limits. Performance evaluation sample results should be plus or minus 10 percent of the true value for metals. See EPA Contract Laboratory Program Statement of Work for Organics Analysis, for organics matrix spike acceptance criteria. Acceptance criteria for organics in the Federal Register, October 26, 1984, 40 CFR Part 136 should also be used to evaluate data.) Chapter 6 of EPA (1979a) provides guidance in identifying problems. A procedure(s) for notifying the Contractor Project Manager who in turn notifies the Lead Agency Project Manager and Ecology Project Officer should also be included in the QA Project Plan (e.g., verbal notification, process for submitting Corrective Action Checklist, Figure 6-1). Acceptance criteria are shown in Table 4-1, Data Quality Objectives for Ground Water Management Areas. Laboratory problems must be resolved before resuming analysis. Samples analyzed while the system was not within acceptable limits must be reanalyzed, if possible.

Reagent and Blank Water

- Organic-free water must be used for all blanks and extractions for organics analysis. Procedures for producing organic-free water and ensuring purity (e.g., preventive maintenance on equipment, etc.) should be specified in the QA Project Plan.
- Deionized, distilled water must be used for all blanks, standards, and any other analytical work related to metals analysis. Procedures for producing deionized, distilled water and ensuring purity (e.g., preventive maintenance on equipment, etc.) should be specified in the QA Project Plan.
- The QA Project Plan should include a description of the reagent water used for analyses other than organics and metals, the purification process used, and techniques to ensure adequate purity.
- Laboratories are encouraged to participate in interlaboratory performance evaluations such as those offered by the U.S. EPA and the American Society for Testing and Materials.

7. DATA REDUCTION, VALIDATION, AND REPORTING

Each QA Project Plan will specify a data flow and reporting scheme that describes individuals responsible for handling data and describes the type of information to be transmitted with the data at each step. In general, independently verified data sets will be provided to the Ecology Project Officer through the Lead Agency Project Manager, with adequate documentation used for independent data validation see Table 7-1 for minimum requirements. The method and level of effort of data validation will be specified in the QA Project Plan, and will be appropriate for the particular task being performed. Criteria for data validation must include checks for internal consistency (e.g., cation/anion balances), checks for transmittal errors, range checks, checks for outliers, checks of laboratory protocol, and checks for overall adherence to the quality control program specified in the QA Project Plan.

Data storage needs unique to the project will be specified after consultation with the Lead Agency Project Manager and Ecology Project Officer and Technical Coordinator. Such needs might include the way in which data are referenced to original documents, particular data qualifiers to be used, the level of detail with which sampling and analysis methods are to be described, results of quality control charts kept by the laboratory, and the inclusion of certain types of descriptive information or additional data. Procedures and criteria for evaluating both historical data and new data for adequacy and suitability for data base inclusion should be developed by the Ecology QA officer in conjunction with the Lead Agency Project Manager and Ecology Project Officer and Technical Coordinator. Any other procedures or criteria used by the grantee for assessing data validity must be described in the QA Project Plan.

The data analysis scheme planned for collected data, including units of measurement, and statistical tests to be used, will be specified in the QA Project Plan. Such descriptions will encourage a well thought out project plan and possibly identify additional data needs (e.g., comparisons of study site data with reference conditions).

Methods for documentation of data analyses performed by the contractor will be stated in the QA Project Plan (e.g., analytical methods used, calibration information, results of quality control samples, copies of pertinent computer printouts), and will be stored with the project data. This will allow personnel performing similar analyses for the same project to examine assumptions and methods and produce comparable results.

Some of the information that should be included in data reports for QA review for organics and inorganics is shown in Tables 7-1 and 7-2 (Tetra Tech, 1986). The QA Project Plan should specify any additional documentation pertinent to the project that will be needed to carry out the independent QA review.

CORRECTIVE ACTION CHECKLIST

Project Name and Number: _____

Sample Dates Involved: _____

Measurement Parameter(s): _____

Acceptable Data Range: _____

Problem Areas Requiring Corrective Action: _____

Measures Required to Correct Problems: _____

Means of Detecting Problems and Verifying Correction: _____

Initiators Name: _____ Date: _____

Project Approval: _____ Date: _____

Laboratory Approval: _____ Date: _____

QA Officer/Reviewer: _____ Date: _____

Figure 6-1. Corrective action checklist.

Table 7-1. Recommended documentation for independent QA review of data on organic substances (from Tetra Tech Inc., 1986).

-
1. Analyses of the requested priority pollutant acids, bases, neutrals (including PCBs and pesticides), and chemically similar compounds should be reported as follows:
 - Sample concentrations reported in proper units (e.g., $\mu\text{g/L}$) to the appropriate number of significant figures on standard data sheets.
 - Lower limits of detection for undetected values reported for each compound on a sample-by-sample basis.
 - Internal standard recoveries for analyses using method recovery standards (including isotope dilution GC/MS), reported on the data sheets as percent recoveries.
 - Ancillary information, including the actual spike level of any recovery standards, final volume of the extract, and injection volume.
 2. Other documentation should include the following:
 - The reconstructed ion chromatogram for each sample (or for each sample fraction if the extract has been analyzed in distinct chemical fractions).
 - GC/ECD chromatograms for pesticide/PCB analyses, with identification of peaks used for quantitation and any confirmation chromatograms.
 - Complete data for all method blanks, reported as absolute mass of each blank contaminant determined; samples associated with each blank should be indicated.
 - Raw data quantitation reports, including tabulated results (identification, GC/MS scan number/retention time, area, and quantity) for compounds in each sample analyzed by GC/MS.
 - A statement in the cover letter describing how standard calibration curves were generated and applied to the samples for quantitation (and access to laboratory records of standard calibration curves for possible inspection).
 - A statement in the cover letter describing any significant problems in any aspect of sample analysis (e.g., instrumental malfunctions, software problems during quantification).
 - A tabulation on standard data sheets of instrument mass detection limits.
-

Table 7-2. Recommended documentation for independent QA review of data on inorganic substances (from Tetra Tech Inc., 1986).

To minimize the amount of backup information provided, only the "raw" instrument readings for the duplicate and spike analyses are requested. Additional backup information would only be required if a review of the QA sample data indicated the need. Data reports from the laboratory should include:

- Sample concentrations reported in proper units to the appropriate number of significant figures
 - Method blank data associated with each sample
 - Quantity of sample digested and final dilution volume
 - Instrument detection limit for each element (denoting method of detection)
 - Method detection limit
 - Summary of all deviations from the prescribed methods
 - Background corrections used (e.g., Zeeman)
 - Spiked sample results with associated calibration procedures and instrument readings
 - Results from all reference materials analyzed with samples
 - All problems associated with the analyses.
-

7.a. Standard Laboratory QA Forms

- The following forms should be used for reporting pertinent analytical and quality control results (forms I - X for organics [except form II]; forms I - VIII for inorganics). Copies of forms and data qualifiers for reporting data are included in the appendix. The mechanism and schedule for transmitting these and other analytical result forms should be included in the QA Project Plan. Copies of all raw data are to be maintained by the laboratory for future review of assumptions and methods for analyzing data.

7.b. Sampling Well Documentation

The following information is to be submitted one time for all wells sampled. It is recommended that the Lead Agency Project Manager as well as the Ecology Project Officer and Technical Coordinator review this information before sampling begins to ensure that sufficient information is available for wells sampled to interpret data for the purpose intended.

- Water Well Report Form showing:
 - location of top of well casing (surveyed both vertically and horizontally)
 - well depth
 - log of materials penetrated with depth
 - construction method, materials (casing, filter pack, grout, etc.)
 - casing and screening material
 - depth and diameter of screened or open interval, screening material
 - type and location of seal
 - owner's name and address
 - driller's name and address
 - results of pump or bailer test(s)
 - if a well has a submersible pump: pump type, pump setting below top of casing, intake location, construction materials, diameter of pump
- Surveyed elevation of the top of the well casing
- Purpose of the well
- Aquifer in which well is completed (if possible)

7.c. Field Data Sheets

Figure 5-1 and Tables 5-3 and 5-4 (or similar forms that include at a minimum data shown on these forms) must be submitted to the Ecology Project Officer (and the Lead Agency Project Manager for each sampling site on each sampling occasion either before the laboratory results are obtained or along with the laboratory results as a single package. Results of laboratory and field quality control check samples and all other available information should be used to interpret laboratory and field results and the data evaluation included in the data report. Schedules for data reports should be outlined in the QA Project Plan.

8. ROUTINE PROCEDURES FOR ASSESSING DATA PRECISION, ACCURACY, AND COMPLETENESS

Intralaboratory precision, accuracy, and completeness will be assessed for each sample batch from duplicate and/or triplicate samples, spike recoveries, and results of standard reference materials analyses. Field analyses should also undergo precision, accuracy, and completeness assessment.

8.a. Precision Assessment

The mean, \bar{C} , of a series of replicate measurements can be calculated by the equation:

$$\bar{C} = (C_1 + C_2 + \dots + C_n)/n$$

where C_1 = the resulting concentration for replicate #1
 C_2 = the resulting concentration for replicate #2, etc.
 n = number of replicate measurements

Precision is estimated for a series of measurements as the coefficient of variation (CV) or relative standard deviation (RSD), where:

$$CV = \frac{\sqrt{\sum_{i=1}^n (C_i - \bar{C})^2}}{\frac{n-1}{\bar{C}}} \times 100\%$$

The precision estimate for duplicate measurements can be expressed as the relative percent difference (RPD):

$$RPD = \frac{C_1 - C_2}{\bar{C}} \times 100\%$$

The CV and RPD values will be compared to respective data quality goals identified in the QA Project Plan.

8.b. Accuracy Assessment

Accuracy is measured via standards, spiked samples, and standard reference material. Replicated samples should make up at least 10 percent of the samples analyzed. The spike recovery accuracy estimate is calculated as:

$$\% \text{ recovery} = \frac{\Delta C}{C_s} \times 100$$

where ΔC = the concentration increase measured due to spiking
(relative to the unspiked portion)
 C_s = the known concentration increase in the spike

Accuracy also will be measured by comparing results of standard reference material analyses with the 95 percent confidence interval for each analyte.

8.c. Completeness Assessment

Completeness is estimated for each set of data by dividing the number of valid analyses (those meeting data quality goals) by the number of samples analyzed. The percent completeness can be described as:

$$\text{Percent completeness} = \frac{\text{Number of Valid Analyses}}{\text{Number of samples analyzed}} \times 100$$

Additional statistical procedures should be used as necessary for estimating data validity. Such methods must be described and references in the QA Project Plan.

8.d. Detection Limit Calculations

For inorganics (metals, nutrients, and major ions), the instrument detection limit is calculated by measuring the variability among method blank measurements. Method blanks are analyzed on three non-consecutive days with seven consecutive measurements per day for the analytes of interest. The average standard deviation of the three-day results is multiplied by three to get the instrument detection limit (EPA Contract Laboratory Program Statement of Work for Metals Analysis). This process should be completed before the laboratory begins analyzing GWMA inorganics samples, and repeated quarterly throughout the project. The instrument detection limit for inorganics (especially trace metals) must be indicated on the data report form.

For organics, the sample detection limit is related to the sample volume, the range of standards used for calibrating the GC, presence of interfering substances, and the blank signal. Laboratories should specify sample detection limits with sample results. Sample detection limits are best determined by analyzing a sample made up of the compounds of interest in the proper solvent. If reliable results are obtained, then the standard solution should be diluted and analyzed again and this process repeated until the level is reached where the analyst cannot confidently distinguish between a real signal and background noise. The method for determining the sample detection limit for organics should be specified in the QA Project Plan.

8.e. Quantitation Limit Calculations

The limit of quantitation for inorganics is calculated by multiplying the standard deviation of the blanks as specified for inorganics under 8.d., above, by 10 (EPA Contract laboratory Program Statement of Work for Metals Analysis).

For organics, the limit of quantitation can be calculated as 10 times the limit of detection as specified for organics in 8.d., above.

The data qualifiers specified with the data reporting forms in the appendix must be used at a minimum to report data. Other data qualifiers may be used in addition to those listed in the appendix to provide more information regarding the reliability of the data, especially a qualifier to label values between the limit of detection and the limit of quantitation. Values in this range have a higher random error than values above the limit of quantitation.

9. PERFORMANCE AND SYSTEM AUDITS

Each Lead Agency QA Project Plan will describe field and laboratory performance system audits to verify the capability of the measurement systems.

Performance and system audits for Lead Agency sampling and analysis operations will consist of on-site reviews of field and laboratory quality assurance procedures and equipment for sampling, calibration, and measurement. Monitoring equipment will be routinely maintained and calibrated during field use (as specified in Tables 5-5 and 6-2 under Preventive Maintenance).

Some analytical laboratories are required to take part in a series of performance and system audits conducted by the National Enforcement Investigations Center (NEIC). For laboratories not involved in these audits, the USEPA Environmental Monitoring Systems/Support Laboratories provide the necessary audit materials, devices, and technical assistance. These laboratories also conduct scheduled interlaboratory performance tests and provide guidance and assistance in the conduct of system audits. Results of these audits are to be made available to the Ecology Project Officer. The following manual for field and laboratory evidence auditing should be used to develop standard procedures for auditing Lead Agency sampling programs:

U.S. EPA, 1981. NEIC Procedures manual for the evidence audit of enforcement investigations by contractor evidence audit teams. EPA-330/9-81-003. National Enforcement Investigations Center, Denver, CO.

The Contractor Project Quality Assurance Coordinator, in conjunction with the Lead Agency Project Manager and Ecology QA Officer, will develop and conduct system audits based on the approved project plan. Performance audits will be conducted soon after the measurement system begins generating data so that problems can be detected before a large amount of unacceptable data is collected. They will be repeated periodically as required by task needs and durations. The Systems Audit Checklist in Figure 9-1 or similar form that includes the information in Figure 9-1 should be used as part of an audit.

Standard quality control solutions used for performance evaluation samples and guidance for their use can be obtained from:

Environmental Monitoring Systems Laboratory. U.S. EPA, Las Vegas, NV 89114

Environmental Monitoring Systems Laboratory. U.S. EPA, Cincinnati, OH 45268

A complete discussion of how to conduct effective field and laboratory system audits is provided in Tetra Tech, Inc. (1985b), Section 6. These procedures (or applicable subset of these) should be implemented in all Lead Agency QA efforts.

10. CORRECTIVE ACTION

Corrective actions can fall into two categories: (1) handling of sampling, analysis, data processing, or equipment failures, and (2) handling of non-conformance or non-compliance with the QA requirements that have been set forth in Table 4-1. Each specific QA Project Plan will address the corrective measures to be taken for the method or equipment failure type of problem. It will include predetermined limits of sample and data acceptability, identification of the corrective action, and the organizational level responsible for approval of action. Routine procedures for correcting and limiting data processing errors will cover data coding, data entry, and data reporting. A formal corrective actions program is difficult to define in advance for the non-conformance or non-compliance type of problems. Each specific QA Project Plan prepared will list who is responsible for taking actions, when actions are to be taken, and who ensures that actions taken produce desired results.

Corrective action will be taken by the Contractor Project Manager when data are found to be outside the predetermined limits of acceptability. The acceptable data range for a specific sampling and analysis task will be listed on the Corrective Actions Checklist (Figure 6-1). The checklist will also provide procedures for implementation of corrective actions.

The QA Project Plan should specify actions to be taken and the person responsible for the action if data quality objectives are not being met. These actions can range from financial penalties, more frequent site visits, meeting with laboratory personnel, or if the situation persists, termination of the contract or grant (Tetra Tech, 1985b).

It is most important that the Ecology Project Officer, Lead Agency Project Manager, and Contractor Project Manager be kept informed of all potentially major quality assurance problems. All three should be notified immediately by telephone of any field or laboratory quality assurance problem that may jeopardize the future use of data collected. The Corrective Actions Checklist also is necessary in such cases, but due to time constraints, is usually not sufficient notification.

11. QUALITY ASSURANCE REPORTS TO MANAGEMENT

Quality assurance reports to the Ecology Project Officer and Lead Agency Project Manager will be made periodically by the Contractor QA coordinator through the Contractor Project Manager. QA Project Plans will describe the mechanism, content, and schedule for the reports. QA reports will be scheduled in relation to completion of various elements of the QA Project Plan rather than to general time periods (e.g., within seven days of each sampling trip).

The reports will compare results regarding data accuracy, precision, and completeness, with the QA Project Plan Objectives for Measurement Data (as in Table 4-1). Reports to management will also include field sampling forms (Figure 5-1 and Tables 5-3 and 5-4), results of system and performance audits, as well as any reports on corrective action, sample alteration, or other significant QA problems. In this manner, effective solutions to project problems may be promptly developed and implemented.

GLOSSARY OF TERMS

AUDIT

A systematic check to determine the quality of operation of some function or activity. Audits may be of two basic types: (1) performance audits on which quantitative data are independently obtained for comparison with routinely obtained data in a measurement system, or (2) system audits of a qualitative nature that consist of an on-site review of a field station laboratory's quality assurance system and physical facilities for sampling, calibrating, and measuring.

CALIBRATION

Checking of physical measurements against accepted standards, including measurements of time, temperature, mass, volume, electrical units, etc. (American Chemical Society Committee on Environmental Improvement, 1980).

DATA QUALITY

The totality of features and characteristics of data that bears on its ability to satisfy a given purpose. The characteristics of major importance are accuracy, precision, completeness, representativeness, and comparability. These characteristics are defined as follows:

Accuracy - the degree of agreement of a measurement (or an average of measurements of the same thing), X , with accepted reference or true value, T , usually expressed as the difference between the two values, $X - T$, or the difference as a percentage of the reference or true value, $100(X - T)/T$, and sometimes expressed as a ratio, X/T . Accuracy is a measure of the bias in the system.

Precision - a measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision is best expressed in terms of the standard deviation. Various measures of precision exist depending upon the "prescribed similar conditions."

Completeness - a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct, normal conditions.

Representativeness - expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

DATA VALIDATION

A systematic process for reviewing a body of data against a set of criteria to provide assurance that the data are adequate for their intended use. Data validation consists of data editing, screening, checking, auditing, verifying, certifying, and reviewing.

DETECTION LIMIT

The lowest concentration of an analyte that the analytical process can reliably detect (ACS Committee on Environmental Improvement, 1980). The

random error at the limit of detection is quite high. For the purposes of Lead Agency sampling programs, the instrument detection limit will be compared to the method detection limit to evaluate acceptability of laboratory performance. (Also see Section 8.d.)

Method Detection Limit - the detection limit specified in the analytical method. Method detection limits for many typical ground water analyses are shown in Table 6-3.

Instrument Detection Limit - the laboratory detection limit based on standard deviations of blank measurements. Each instrument in the laboratory has a unique instrument detection limit. See 8.d. for calculation of instrument detection limits.

QUALITY ASSURANCE

The total integrated program for assuring the reliability of monitoring and measurement data. A system for integrating the quality planning, quality assessment, and quality improvement efforts to meet user requirements.

QUALITY ASSURANCE PROGRAM PLAN

An orderly assemblage of management policies, objectives, principles, and general procedures by which an agency or laboratory outlines how it intends to produce data of known and accepted quality.

QUALITY ASSURANCE PROJECT PLAN

An orderly assemblage of detailed and specific procedures which delineates how data of known and acceptable quality are produced for a specific project. (A given agency or laboratory would have only one quality assurance plan, but would have a specific Quality Assurance Project Plan [QA Project Plan] for each of its projects.)

QUALITY CONTROL

The routine application of procedures for obtaining prescribed standards of performance in the monitoring and measurement process.

QUANTITATION LIMIT

The range of concentration of an analyte that is clearly above the detection limit. The random error of values above the quantitation limit is lower than for values between the detection limit and the quantitation limit. (Also see Section 8.d.)

REFERENCES

- Barcelona, M.J., 1985. Material from Monitoring the Saturated and Unsaturated Zones, National Water Well Association, April 1985, Seattle, WA.
- Barcelona, M.J., J.P. Gibb, J.A. Helfrich, E.E. Garske, 1985a. Practical Guide for Ground water Sampling. Prepared in cooperation with EPA EMSL, Las Vegas, NV, by Illinois State Water Survey. SWS Contract Report 374. 94 pp.
- Barcelona, M.J., J.A. Helfrich, and E.E. Garske, 1985b. Sampling tubing effects on ground water samples. *Analytical Chemistry* 57(2): 460-464. (Errata page: *Analytical Chemistry* 57(13): 2752.
- Barcelona, M.J., J.A. Helfrich, E.E. Garske, and J.P. Gibb, 1984. A Laboratory evaluation of ground water sampling mechanisms. *Ground Water Monitoring Review* 4(2): 32-41.
- Claassen, H.C., 1982. Guidelines and techniques for obtaining water samples that accurately represent the water chemistry of an aquifer. U.S. Geological Survey Open File Report 82-1024. Lakewood, CO, 49 pp.
- Electric Power Research Institute, 1985. Preliminary results on chemical changes in groundwater samples due to sampling devices. EPRI EA-4118, Project 2485-7. Prepared by Residuals Management Technology, Inc., Madison, WI.
- Garske, E.E. and M.R. Schock, 1986. An inexpensive flow-through cell and measurement system for monitoring selected chemical parameters in ground water. *Ground Water Monitoring Review* 6(3): 79-84.
- Gibb, J.P., R.M. Schuller, and R.A. Griffin, 1981. Procedures for the collection of representative water quality data from monitoring wells. Illinois State Water Survey/Geological Survey.
- Gibb, J.P., 1985. Sampling procedures for monitoring wells. Section 11 of course notes from NWWA short course: Ground Water and Unsaturated Zones, Monitoring and Sampling. Seattle, WA. April 30 - May 2, 1985.
- Golder Associates, Inc., 1986. Quality assurance (QA) project plan for remedial investigations (RI) at the North Market Street site. Prepared for the Washington State Department of Ecology.
- NCASI (National Council of the Paper Industry for Air and Stream Improvement), 1982. A Guide to Groundwater Sampling, Technical Bulletin No. 362.
- New Jersey Department of Environmental Protection, Division of Water Resources, 1983. Field Procedures Manual for Water Quality Acquisition, Office of Quality Assurance, Trenton, NJ, 440 pp. plus appendices.
- Papadopoulos, I.S. and H. Cooper, 1967. Drawdown in a well of large diameter. *Water Resources Research*, 3(1): 241-244.
- Rajagopal, R., 1986. The effect of sampling frequency on ground water quality characterization. *Ground water Monitoring Review*. Fall. 6(4): 65-73.
- Schmidt, K.D., 1985. How representative are water samples collected from wells? From course notes from NWWA short course: Ground Water and Unsaturated Zones Monitoring and Sampling, Seattle WA. April 30 - May 2, 1985.

Tetra Tech, Inc., 1986. Quality Assurance Project Plan for Field Investigations to Support Commencement Bay Nearshore/Tideflats Feasibility Study. Prepared for Washington State Department of Ecology.

Tetra Tech, Inc., 1985a. Draft quality assurance program plan for remedial investigations. Prepared for Black and Veatch and Washington State Department of Ecology.

Tetra Tech, Inc., 1985b. Final Report. Quality assurance management plan for remedial investigations. Prepared for Black and Veatch and Washington State Department of Ecology.

Tetra Tech, Inc., 1985c. Remedial action division data management recommended approaches. Draft report prepared for Black and Veatch and Washington State Department of Ecology.

U.S. EPA. User's Guide to the Contract Laboratory Program, Sample Management Office, Washington, D.C.

U.S. EPA, Region 10, Quality Assurance Management Office, 1986. Quality Assurance Manual for Waste Management Branch Investigations.

U.S. EPA, 1985. RCRA Ground water Monitoring Technical Enforcement Guidance Document (draft).

U.S. EPA, 1984. Calculation of precision, bias, and method detection limit for chemical and physical measurements. Office of Monitoring Systems and Quality Assurance, Office of Research and Development, Washington, D.C.

U.S. EPA, 1982. Test Methods for Evaluating Solid Waste. SW-846. Office of Solid Waste.

U.S. EPA, 1979a. Handbook for Analytical Quality Control in Water and Wastewater Laboratories. EPA-600/4-79-019. Environmental Monitoring and Support Laboratory, Cincinnati, OH.

U.S. EPA, 1979b. Quality Assurance Guidelines for IERL-C: Project Officers. EPA-600/9-79-046. Industrial Environmental Research Laboratory, Cincinnati, OH.

U.S. EPA, 1979c. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020. EMSL, Cincinnati, OH.

U.S. Federal Register, October 26, 1984. EPA Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule. 40 CFR Part 136. 210 pp.

→ U.S. Geological Survey, 1980. Handbook of Recommended Methods for Water Data Acquisition, Reston, VA.

APPENDIX

ORGANIC DATA REPORTING FORMS

DATA REPORTING QUALIFIERS - ORGANICS

For reporting results to Ecology, the following results qualifiers are used. Additional flags or footnotes explaining results are encouraged. However, the definition of such flags must be explicit.

- Value - If the result is a value greater than or equal to the detection limit, report the value.
- U - Indicates the compound was analyzed for but not detected. Report the minimum detection limit for the sample with the U (e.g., 10U) based on necessary concentration/dilution actions. (This is not necessarily the instrument detection limit.) The footnote should read: U-Compound was analyzed for but not detected. The number is the minimum attainable detection limit for the sample.
- J - Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed or when the mass spectral data indicate the presence of a compound that meets the identification criteria but the result is less than the specified detection limit but greater than zero (e.g., 10J).
- C - This flag applies to pesticide parameters where the identification has been confirmed by GC/MS. Single component pesticides greater than or equal to 10 ng/ul in the final extract should be confirmed by GC/MS.
- B - This flag is used when the analyte is found in the blank as well as a sample. It indicates possible/probable blank contamination and warns the data user to take appropriate action.
- Other - Other specific flags and footnotes may be required to properly define the results. If used, they must be fully described and such description attached to the data summary report.

DATA REPORTING QUALIFIERS - INORGANICS

- Value - If the result is a value greater than or equal to the instrument detection limit but less than the contract required detection limit, report the value in brackets (i.e., [10]). Indicate the analytical method used with P (for ICP/Flame AA) or F (for furnace).
 - U - Indicates element was analyzed for but not detected. Report with the detection limit value (e.g., 10U).
 - E - Indicates a value estimated or not reported due to the presence of interference. Explanatory note included on cover page.
 - s - Indicates value determined by Method of Standard Addition.
 - R - Indicates spike sample recovery is not within control limits.
 - * - Indicates duplicate analysis is not within control limits.
 - +
- Indicates the correlation coefficient for method of standard addition is less than 0.995.

SYSTEMS AUDIT CHECKLIST

Sample Program Identification: _____

Sampling Dates: _____

Material to be Sampled: _____

Measurement Parameter: _____

Sampling and Monitoring Equipment in Use:

_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

Audit Procedures and Frequency: _____

Field or Laboratory Calibration Procedures and Frequency: _____

Signature of QA Coordinator: _____ Date: _____

Figure 9-1. System audit checklist.

Organics Analysis Data Sheet (Page 1)

Laboratory Name: _____

Case No: _____

Lab Sample ID No: _____

QC Report No: _____

Sample Matrix: _____

Contract No: _____

Data Release Authorized By: _____

Date Sample Received: _____

Volatile Compounds

Concentration: Low Medium (Circle One)

Date Extracted/Prepared: _____

Date Analyzed: _____

Conc/Dil Factor: _____ pH _____

Percent Moisture: (Not Decanted) _____

CAS Number		ug/l or ug/Kg (Circle One)
74-87-3	Chloromethane	
74-83-9	Bromomethane	
75-01-4	Vinyl Chloride	
75-00-3	Chloroethane	
75-09-2	Methylene Chloride	
67-64-1	Acetone	
75-15-0	Carbon Disulfide	
75-35-4	1, 1-Dichloroethene	
75-34-3	1, 1-Dichloroethane	
156-60-5	Trans-1, 2-Dichloroethene	
67-66-3	Chloroform	
107-06-2	1, 2-Dichloroethane	
78-93-3	2-Butanone	
71-55-6	1, 1, 1-Trichloroethane	
56-23-5	Carbon Tetrachloride	
108-05-4	Vinyl Acetate	
75-27-4	Bromodichloromethane	

CAS Number		ug/l or ug/Kg (Circle One)
78-87-5	1, 2-Dichloropropane	
10061-02-6	Trans-1, 3-Dichloropropene	
79-01-6	Trichloroethene	
124-48-1	Dibromochloromethane	
79-00-5	1, 1, 2-Trichloroethane	
71-43-2	Benzene	
10061-01-5	cis-1, 3-Dichloropropene	
110-75-8	2-Chloroethylvinylether	
75-25-2	Bromoform	
108-10-1	4-Methyl-2-Pentanone	
591-78-6	2-Hexanone	
127-18-4	Tetrachloroethene	
79-34-5	1, 1, 2, 2-Tetrachloroethane	
108-88-3	Toluene	
108-90-7	Chlorobenzene	
100-41-4	Ethylbenzene	
100-42-5	Styrene	
	Total Xylenes	

Data Reporting Qualifiers

For reporting results to EPA the following results qualifiers are used
Additional flags or footnotes explaining results are encouraged. However, the
definition of each flag must be explicit

Value If the result is a value greater than or equal to the detection limit report the value.

U Indicates compound was analyzed for but not detected. Report the minimum detection limit for the sample with the U (e.g., 10U) based on necessary concentration/dilution action. (This is not necessarily the instrument detection limit.) The footnote should read: U-Compound was analyzed for but not detected. The number is the minimum attainable detection limit for the sample.

J Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed or when the mass spectral data indicated the presence of a compound that meets the identification criteria but the result is less than the specified detection limit but greater than zero. (e.g., 10J). If limit of detection is 10 µg/l and a concentration of 3 µg/l is calculated, report as 3J.

C This flag applies to pesticide parameters where the identification has been confirmed by GC/MS. Single component pesticides ≥ 10 ng/ul in the final extract should be confirmed by GC/MS.

B This flag is used when the analyte is found in the blank as well as a sample. It indicates possible/probable blank contamination and warns the data user to take appropriate action.

Other Other specific flags and footnotes may be required to properly define the results. If used, they must be fully described and such description attached to the data summary report.

Laboratory Name _____

Case No: _____

Sample Number

Organics Analysis Data Sheet (Page 2)

Semivolatile Compounds

Concentration: Low Medium (Circle One)

Date Extracted / Prepared: _____

Date Analyzed: _____

Conc / Dil Factor: _____

Percent Moisture (Decanted): _____

GPC Cleanup ☐ Yes ☐ NoSeparatory Funnel Extraction ☐ YesContinuous Liquid - Liquid Extraction ☐ Yes

CAS Number		ug / l or ug / Kg (Circle One)
108-95-2	Phenol	
111-44-4	bis(2-Chloroethyl)Ether	
95-57-8	2-Chlorophenol	
541-73-1	1, 3-Dichlorobenzene	
106-46-7	1, 4-Dichlorobenzene	
100-51-6	Benzyl Alcohol	
95-50-1	1, 2-Dichlorobenzene	
95-48-7	2-Methylphenol	
39638-32-9	bis(2-chloroisopropyl)Ether	
106-44-5	4-Methylphenol	
621-64-7	N-Nitroso-Di-n-Propylamine	
67-72-1	Hexachloroethane	
98-95-3	Nitrobenzene	
78-59-1	Isophorone	
88-75-5	2-Nitrophenol	
105-67-9	2, 4-Dimethylphenol	
65-85-0	Benzoic Acid	
111-91-1	bis(2-Chloroethoxy)Methane	
120-83-2	2, 4-Dichlorophenol	
120-82-1	1, 2, 4-Trichlorobenzene	
91-20-3	Naphthalene	
106-47-8	4-Chloroaniline	
87-68-3	Hexachlorobutadiene	
59-50-7	4-Chloro-3-Methylphenol	
91-57-6	2-Methylnaphthalene	
77-47-4	Hexachlorocyclopentadiene	
88-06-2	2, 4, 6-Trichlorophenol	
95-95-4	2, 4, 5-Trichlorophenol	
91-58-7	2-Chloronaphthalene	
88-74-4	2-Nitroaniline	
131-11-3	Dimethyl Phthalate	
208-96-8	Acenaphthylene	
99-09-2	3-Nitroaniline	

CAS Number		ug / l or ug / Kg (Circle One)
83-32-9	Acenaphthene	
51-28-5	2, 4-Dinitrophenol	
100-02-7	4-Nitrophenol	
132-64-9	Dibenzofuran	
121-14-2	2, 4-Dinitrotoluene	
606-20-2	2, 6-Dinitrotoluene	
84-66-2	Diethylphthalate	
7005-72-3	4-Chlorophenyl-phenylether	
86-73-7	Fluorene	
100-01-6	4-Nitroaniline	
534-52-1	4, 6-Dinitro-2-Methylphenol	
86-30-6	N-Nitrosodiphenylamine (1)	
101-55-3	4-Bromophenyl-phenylether	
118-74-1	Hexachlorobenzene	
87-86-5	Pentachlorophenol	
85-01-8	Phenanthrene	
120-12-7	Anthracene	
84-74-2	Di-n-Butylphthalate	
206-44-0	Fluoranthene	
129-00-0	Pyrene	
85-68-7	Butylbenzylphthalate	
91-94-1	3, 3'-Dichlorobenzidine	
56-55-3	Benzo(a)Anthracene	
117-81-7	bis(2-Ethylhexyl)Phthalate	
218-01-9	Chrysene	
117-84-0	Di-n-Octyl Phthalate	
205-99-2	Benzo(b)Fluoranthene	
207-08-9	Benzo(k)Fluoranthene	
50-32-8	Benzo(a)Pyrene	
193-39-5	Indeno(1, 2, 3-cd)Pyrene	
53-70-3	Dibenz(a, h)Anthracene	
191-24-2	Benzo(g, h, i)Perylene	

(1) Cannot be separated from diphenylamine

Laboratory Name _____

Case No _____

Sample Number

Organics Analysis Data Sheet (Page 3)

Pesticide/PCBs

Concentration Low Medium (Circle One)

GPC Cleanup ☐ Yes ☐ No

Date Extracted / Prepared _____

Separatory Funnel Extraction ☐ Yes

Date Analyzed _____

Continuous Liquid - Liquid Extraction ☐ Yes

Conc / Dil Factor _____

Percent Moisture (decanted) _____

CAS Number		ug/l or ug/Kg (Circle One)
319-84-6	Alpha BHC	
319-85-7	Beta-BHC	
319-86-8	Delta-BHC	
58-89-9	Gamma-BHC (Lindane)	
76-44-8	Heptachlor	
309-00-2	Aldrin	
1024-57-3	Heptachlor Epoxide	
959-98-8	Endosulfan I	
60-57-1	Dieldrin	
72-55-9	4, 4'-DDE	
72-20-8	Endrin	
33213-65-9	Endosulfan II	
72-54-8	4, 4'-DDD	
1031-07-8	Endosulfan Sulfate	
50-29-3	4, 4'-DDT	
72-43-5	Methoxychlor	
53494-70-5	Endrin Ketone	
57-74-9	Chlordane	
8001-35-2	Toxaphene	
12674-11-2	Aroclor-1016	
11104-28-2	Aroclor-1221	
11141-16-5	Aroclor-1232	
53469-21-9	Aroclor-1242	
12672-29-6	Aroclor-1248	
11097-69-1	Aroclor-1254	
11096-82-5	Aroclor-1260	

 V_i = Volume of extract injected (ul) V_s = Volume of water extracted (ml) W_s = Weight of sample extracted (g) V_t = Volume of total extract (ul) V_s _____ or W_s _____ V_t _____ V_i _____

Laboratory Name _____

Case No _____

Sample Number

Organics Analysis Data Sheet
(Page 4)

Tentatively Identified Compounds

CAS Number	Compound Name	Fraction	RT or Scan Number	Estimated Concentration (ug/l or ug/kg)
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				
27				
28				
29				
30				

Case No. _____ Contract Laboratory _____ Contract No. _____

* VALUES ARE OUTSIDE OF CONTRACT REQUIRED QC LIMITS

** ADVISORY LIMITS ONLY

Volatiles:	_____	out of _____	_____	outside of QC limits
Semi-Volatiles:	_____	out of _____	_____	outside of QC limits
Pesticides:	_____	out of _____	_____	outside of QC limits

7185

SOIL SURROGATE PERCENT RECOVERY SUMMARY

Case No. _____ Contract Laboratory _____ Contract No. _____

Low _____ Medium _____

[illegible]

* VALUES ARE OUTSIDE OF CONTRACT REQUIRED QC LIMITS

****ADVISORY LIMITS ONLY**

Volatiles: _____ out of _____; outside of QC limits

Semi-Volatiles: _____ out of _____; outside of QC limits

Pesticides: _____ out of _____ ; outside of QC limits

Comments:

FORM II

WATER MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Case No. _____ Contractor _____ Contract No. _____

FRACTION	COMPOUND	CONC. SPIKE ADDED (ug/L)	SAMPLE RESULT	CONC. MS	% REC	CONC. MSD	% REC	RPD	QC LIMITS * RECOVERY
VOA SMO SAMPLE NO.	1,1-Dichloroethene								14 61-145
	Trichloroethene								14 71-120
	Chlorobenzene								13 75-130
	Toluene								13 76-125
	Benzene								11 78-127
B/N SMO SAMPLE NO.	1,2,4-Trichlorobenzene								28 39-98
	Acenaphthene								31 46-118
	2,4 Dinitrotoluene								38 24-96
	Pyrene								31 26-127
	N-Nitroso-Di-n-Propylamine								38 41-116
ACID SMO SAMPLE NO.	1,4-Dichlorobenzene								28 36-97
	Pentachlorophenol								50 9-103
	Phenol								42 12-89
	2-Chlorophenol								40 27-123
	4-Chloro-3-Methylphenol								42 23-97
PEST SMO SAMPLE NO.	4-Nitrophenol								50 10-80
	Lindane								15 56-123
	Heptachlor								20 40-131
	Aldrin								22 40-120
	Dieldrin								18 52-126
	Endrin								21 56-121
	4,4'-DDT								27 38-127

* ASTERISKED VALUES ARE OUTSIDE QC LIMITS.

RPD: VOAs _____ out of _____ outside QC limits
 B/N _____ out of _____ outside QC limits
 ACID _____ out of _____ outside QC limits
 PEST _____ out of _____ outside QC limits

RECOVERY: VOAs _____ out of _____ outside QC limits
 B/N _____ out of _____ outside QC limits
 ACID _____ out of _____ outside QC limits
 PEST _____ out of _____ outside QC limits

Comments: _____

SOIL MATRIX SPIKE /MATRIX SPIKE DUPLICATE RECOVERY

Case No. _____ Contractor _____ Contract No. _____

Low Level _____ Medium Level _____

FRACTION	COMPOUND	CONC. SPIKE ADDED (ug/Kg)	SAMPLE RESULT	CONC. MS	% REC	CONC. MSD	% REC	RPD	QC LIMITS RECOVERY
VOA SMO SAMPLE NO.	1,1-Dichloroethene								22 59-172
	Trichloroethene								24 62-137
	Chlorobenzene								21 60-133
	Toluene								21 59-139
	Benzene								21 66-142
B/N SMO SAMPLE NO.	1,2,4-Trichlorobenzene								23 38-107
	Acenaphthene								19 31-137
	2,4 Dinitrotoluene								47 28-89
	Pyrene								36 35-142
	N-Nitrosodi-n-Propylamine								38 41-126
ACID SMO SAMPLE NO.	1,4-Dichlorobenzene								27 28-104
	Pentachlorophenol								47 17-109
	Phenol								35 26-90
	2-Chlorophenol								50 25-102
	4-Chloro-3-Methylphenol								33 26-103
PEST SMO SAMPLE NO.	4-Nitrophenol								50 11-114
	Lindane								50 46-127
	Heptachlor								31 35-130
	Aldrin								43 34-132
	Dieldrin								38 31-134
	Endrin								45 42-139
	4,4'-DDT								50 23-134

*ASTERISKED VALUES ARE OUTSIDE QC LIMITS.

RPD: VOAs _____ out of _____ ; outside QC limits
 B/N _____ out of _____ ; outside QC limits
 ACID _____ out of _____ ; outside QC limits
 PEST _____ out of _____ ; outside QC limits

RECOVERY: VOAs _____ out of _____ ; outside QC limits
 B/N _____ out of _____ ; outside QC limits
 ACID _____ out of _____ ; outside QC limits
 PEST _____ out of _____ ; outside QC limits

Comments: _____

METHOD BLANK SUMMARY

Case No. _____ Region _____ Contractor _____ Contract No. _____

[illegible]**Comments:**

FORM IV

7185

Case No. _____ Contractor _____ Contract No. _____
Instrument ID _____ Date _____ Time _____
Lab ID _____ Data Release Authorized By: _____

m/e	ION ABUNDANCE CRITERIA	%RELATIVE ABUNDANCE
50	15.0 - 40.0% of the base peak	
75	30.0 - 60.0% of the base peak	
95	Base peak, 100% relative abundance	
96	5.0 - 9.0% of the base peak	
173	Less than 1.0% of the base peak	
174	Greater than 50.0% of the base peak	
175	5.0 - 9.0% of mass 174	() ¹
176	Greater than 95.0% but less than 101.0% of mass 174	() ¹
177	5.0 - 9.0% of mass 176	() ²

¹ Value in parenthesis is % mass 174²Value in parenthesis is % mass 176[illegible]

Case No. _____ Contractor _____ Contract No. _____
Instrument ID _____ Date _____ Time _____
Lab ID _____ Data Release Authorized By: _____

THIS PERFORMANCE TUNE APPLIES TO THE FOLLOWING
SAMPLES, BLANKS AND STANDARDS.

²Value in parenthesis is % mass 442

FORM V

Initial Calibration Data Volatile HSL Compounds

Case No: _____ Region: _____ Instrument I D: _____
 Contractor: _____ Calibration Date: _____
 Contract No: _____

Minimum RF for SPCC is 0.300
 (0.25 for Bromoform)

Maximum % RSD for CCC is 30%

Laboratory ID								
Compound	RF ₂₀	RF ₅₀	RF ₁₀₀	RF ₁₅₀	RF ₂₀₀	RF	% RSD	CCC- SPCC**
Chloromethane								* *
Bromomethane								
Vinyl Chloride								*
Chloroethane								
Methylene Chloride								
Acetone								
Carbon Disulfide								
1, 1-Dichloroethene								*
1, 1-Dichloroethane								* *
Trans-1, 2-Dichloroethene								
Chloroform								*
1, 2-Dichloroethane								
2-Butanone								
1, 1, 1-Trichloroethane								
Carbon Tetrachloride								
Vinyl Acetate								
Bromodichloromethane								
1, 2-Dichloropropane								*
Trans-1, 3-Dichloropropene								
Trichloroethene								
Dibromochloromethane								
1, 1, 2-Trichloroethane								
Benzene								
cis-1, 3-Dichloropropene								
2-Chloroethylvinylether								
Bromoform								* *
4-Methyl-2-Pentanone								
2-Hexanone								
Tetrachloroethene								
1, 1, 2, 2-Tetrachloroethane								* *
Toluene								*
Chlorobenzene								* *
Ethylbenzene								*
Styrene								
Total Xylenes								

RF - Response Factor (subscript is the amount of ug/L)
 RF - Average Response Factor
 %RSD - Percent Relative Standard Deviation

CCC - Calibration Check Compounds (*)
 SPCC - System Performance Check Compounds (**)

Form VI

Initial Calibration Data
Semivolatile HSL Compounds
(Page 1)

Case No: _____ Region: _____ Instrument ID: _____
Contractor: _____ Calibration Date: _____
Contract No: _____

Minimum \overline{RF} for SPCC is 0.050 Maximum % RSD for CCC is 30%

Laboratory ID								
Compound	RF ₂₀	RF ₅₀	RF ₈₀	RF ₁₂₀	RF ₁₆₀	\overline{RF}	% RSD	CCC• SPCC**
Phenol								*
bis(-2-Chloroethyl)Ether								
2-Chlorophenol								
1, 3-Dichlorobenzene								
1, 4-Dichlorobenzene								*
Benzyl Alcohol								
1, 2-Dichlorobenzene								
2-Methylphenol								
bis(2-chloroisopropyl)Ether								
4-Methylphenol								
N-Nitroso-Di-n-Propylamine								* *
Hexachloroethane								
Nitrobenzene								
Isophorone								
2-Nitrophenol								*
2, 4-Dimethylphenol								
Benzoic Acid	†							
bis(-2-Chloroethoxy)Methane								
2, 4-Dichlorophenol								
1, 2, 4-Trichlorobenzene								
Naphthalene								
4-Chloroaniline								
Hexachlorobutadiene								*
4-Chloro-3-Methylphenol								*
2-Methylnaphthalene								
Hexachlorocyclopentadiene								* *
2, 4, 6-Trichlorophenol								*
2, 4, 5-Trichlorophenol	†							
2-Chloronaphthalene								
2-Nitroaniline	†							
Dimethyl Phthalate								
Acenaphthylene								
3-Nitroaniline	†							
Acenaphthene								*
2, 4-Dinitrophenol	†							* *
4-Nitrophenol	†							* *
Dibenzofuran								

Response Factor (subscript is the amount of nanograms)

\overline{RF} - Average Response Factor

%RSD - Percent Relative Standard Deviation

CCC -Calibration Check Compounds (*)

SPCC -System Performance Check Compounds (**)

† -Not detectable at 20 ng

**Initial Calibration Data
Semivolatile HSL Compounds
(Page 2)**

Case No: _____ Region: _____ Instrument ID: _____
 Contractor: _____ Calibration Date: _____
 Contract No: _____

Minimum \overline{RF} for SPCC is 0.050 Maximum % RSD for CCC is 30%

Laboratory ID								
Compound	RF ₂₀	RF ₅₀	RF ₈₀	RF ₁₂₀	RF ₁₆₀	\overline{RF}	% RSD	CCC- SPCC-*
2, 4-Dinitrotoluene								
2, 6-Dinitrotoluene								
Diethylphthalate								
4-Chlorophenyl-phenylether								
Fluorene								
4-Nitroaniline	†							
4, 6-Dinitro-2-Methylphenol	†							
N-Nitrosodiphenylamine (1)								*
4-Bromophenyl-phenylether								
Hexachlorobenzene								
Pentachlorophenol	†							*
Phenanthrene								
Anthracene								
Di-N-Butylphthalate								
Fluoranthene								*
Pyrene								
Butylbenzylphthalate								
3, 3'-Dichlorobenzidine								
Benzo(a)Anthracene								
bis(2-Ethylhexyl)Phthalate								
Chrysene								
Di-n-Octyl Phthalate								*
Benzo(b)Fluoranthene								
Benzo(k)Fluoranthene								
Benzo(a)Pyrene								*
Indeno(1, 2, 3-cd)Pyrene								
Dibenz(a, h)Anthracene								
Benzo(g, h, i)Perylene								

Response Factor (subscript is the amount of nanograms)
 \overline{RF} -Average Response Factor
 %RSD -Percent Relative Standard Deviation
 CCC -Calibration Check Compounds (*)

SPCC -System Performance Check Compounds (**)
 † -Not detectable at 20 ng
 (1) -Cannot be separated from diphenylamine

Form VI

**Continuing Calibration Check
Semivolatile HSL Compounds
(Page 1)**

Case No: _____ Region: _____ Calibration Date: _____
 Contractor: _____ Time: _____
 Contract No: _____ Laboratory ID: _____
 Instrument ID: _____ Initial Calibration Date: _____

Minimum RF for SPCC is 0.050 Maximum %D for CCC is 25%

Compound	RF	RF ₅₀	% D	CCC	SPCC
Phenol				*	
bis(2-Chloroethyl)Ether					
2-Chlorophenol					
1, 3-Dichlorobenzene					
1, 4-Dichlorobenzene				*	
Benzyl Alcohol					
1, 2-Dichlorobenzene					
2-Methylphenol					
bis(2-chloroisopropyl)Ether					
4-Methylphenol					
N-Nitroso-Di-n-Propylamine					* *
Hexachloroethane					
Nitrobenzene					
Isophorone					
2-Nitrophenol				*	
2, 4-Dimethylphenol					
Benzoic Acid †					
bis(2-Chloroethoxy)Methane					
2, 4-Dichlorophenol				*	
1, 2, 4-Trichlorobenzene					
Naphthalene					
4-Chloroaniline					
Hexachlorobutadiene				*	
4-Chloro-3-Methylphenol				*	
2-Methylnaphthalene					
Hexachlorocyclopentadiene					* *
2, 4, 6-Trichlorophenol				*	
2, 4, 5-Trichlorophenol †					
2-Chloronaphthalene					
2-Nitroaniline †					
Dimethyl Phthalate					
Acenaphthylene					
3-Nitroaniline †					
Acenaphthene				*	
2, 4-Dinitrophenol †					* *
4-Nitrophenol †					* *
Dibenzofuran					

RF₅₀ Response Factor from daily standard file at concentration indicated (50 total nanograms)

RF Average Response Factor from initial calibration Form VI

† -Due to low response, analyze at 80 total nanograms

%D Percent Difference

CCC Calibration Check Compounds (-)

SPCC System Performance Check Compounds (-)

**Continuing Calibration Check
Semivolatile HSL Compounds
(Page 2)**

Case No: _____ Region: _____

Calibration Date: _____

Contractor: _____

Time: _____

Contract No: _____

Laboratory ID: _____

Instrument ID: _____

Initial Calibration Date: _____

Minimum RF for SPCC is 0.050

Maximum %D for CCC is 25%

Compound	RF	RF ₅₀	% D	CCC	SPCC
2, 4-Dinitrotoluene					
2, 6-Dinitrotoluene					
Diethylphthalate					
4-Chlorophenyl-phenylether					
Fluorene					
4-Nitroaniline †					
4, 6-Dinitro-2-Methylphenol †					
N-Nitrosodiphenylamine (1)				*	
4-Bromophenyl-phenylether					
Hexachlorobenzene					
Pentachlorophenol †				*	
Phenanthrene					
Anthracene					
Di-N-Butylphthalate					
Fluoranthene				*	
Pyrene					
Butylbenzylphthalate					
3, 3'-Dichlorobenzidine					
Benzo(a)Anthracene					
bis(2-Ethylhexyl)Phthalate					
Chrysene					
Di-n-Octyl Phthalate				*	
Benzo(b)Fluoranthene					
Benzo(k)Fluoranthene					
Benzo(a)Pyrene				*	
Indeno(1, 2, 3-cd)Pyrene					
Dibenz(a, h)Anthracene					
Benzo(g, h, i)Perylene					

RF₅₀ -Response Factor from daily standard file at concentration indicated (50 total nanograms)

RF -Average Response Factor from initial calibration Form VI

%D -Percent Difference

† -Due to low response analyze at 80 total nanograms

CCC -Calibration Check Compounds (---)

SPCC -System Performance Check Compounds (---)

(1) Cannot be separated from diphenylamine

Form VII

Continuing Calibration Check Volatile HSL Compounds

Case No: _____ Region: _____
Contractor: _____
Contract No: _____
Instrument ID: _____

Calibration Date: _____
Time: _____
Laboratory ID: _____
Initial Calibration Date: _____

Minimum RF for SPCC is 0.300
(0.25 for Bromoform)

Maximum %D for CCC is 25%

Compound	RF	RF ₅₀	% D	CCC	SPCC
Chloromethane					* *
Bromomethane					
Vinyl Chloride				*	
Chloroethane					
Methylene Chloride					
Acetone					
Carbon Disulfide					
1, 1-Dichloroethene				*	
1, 1-Dichloroethane					* *
Trans-1, 2-Dichloroethene					
Chloroform				*	
1, 2-Dichloroethane					
2-Butanone					
1, 1, 1-Trichloroethane					
Carbon Tetrachloride					
Vinyl Acetate					
Bromodichloromethane					
1, 2-Dichloropropane				*	
Trans-1, 3-Dichloropropene					
Trichloroethene					
Dibromochloromethane					
1, 1, 2-Trichloroethane					
Benzene					
cis-1, 3-Dichloropropene					
2-Chloroethylvinylether					
Bromoform					* *
4-Methyl-2-Pentanone					
2-Hexanone					
Tetrachloroethene					
1, 1, 2, 2-Tetrachloroethane					* *
Toluene				*	
Chlorobenzene					* *
Ethylbenzene				*	
Styrene					
Total Xylenes					

RF₅₀ - Response Factor from daily standard file at 50 ug/l
RF - Average Response Factor from initial calibration Form VI

%D - Percent Difference
CCC - Calibration Check Compounds (*)
SPCC - System Performance Check Compounds (**)

Form VII

Pesticide Evaluation Standards Summary
(Page 1)

Case No. _____ Region: _____
Contract No: _____
Date of Analysis: _____

Laboratory: _____
GC Column: _____
Instrument ID: _____

Evaluation Check for Linearity

Laboratory ID				
Pesticide	Calibration Factor Eval Mix A	Calibration Factor Eval Mix B	Calibration Factor Eval Mix C	% RSD (≤ 10%)
Aldrin				
Endrin				
4,4'-DDT ⁽¹⁾				
Dibutyl Chlorendate				

Evaluation Check for 4,4'-DDT/Endrin Breakdown
(percent breakdown expressed as total degradation)

	Laboratory ID	Time of Analysis	Endrin	4,4'-DDT	Combined ⁽²⁾
Eval Mix B 72 Hour					
Eval Mix B					
Eval Mix B					
Eval Mix B					
Eval Mix B					
Eval Mix B					
Eval Mix B					
Eval Mix B					
Eval Mix B					
Eval Mix B					
Eval Mix B					

(1) See Exhibit E, Section 7.5.4

(2) See Exhibit E, Section 7.3.1.2.2.1

(Page 2)

Report all standards, blanks and samples

[illegible]

PESTICIDE/PCB STANDARDS SUMMARY

Case No. _____ Laboratory _____ GC Column _____ GC Instrument ID _____
 Contract No. _____

COMPOUND	DATE OF ANALYSIS _____ TIME OF ANALYSIS _____ LABORATORY ID _____			DATE OF ANALYSIS _____ TIME OF ANALYSIS _____ LABORATORY ID _____			RT	CALIBRATION FACTOR	CONF. OR QUANT.	CALIBRATION FACTOR	CONF. OR QUANT.	PERCENT DIFF. **
	RT	RETENTION TIME WINDOW	CALIBRATION FACTOR	CONF. OR QUANT.	RT	CALIBRATION FACTOR						
alpha - BHC												
beta - BHC												
delta - BHC												
gamma - BHC												
Heptachlor												
Aldrin												
Heptachlor Epoxide												
Endosulfan I												
Dieldrin												
4,4'-DDE												
Endrin												
Endosulfan II												
4,4'-DDD												
Endosulfan Sulfate												
4,4'-DDT												
Methoxychlor												
Endrin Ketone												
Tech. Chlordane												
alpha - Chlordane*												
gamma - Chlordane*												
Toxaphene												
Aroclor - 1016												
Aroclor - 1221												
Aroclor - 1232												
Aroclor - 1242												
Aroclor - 1248												
Aroclor - 1254												
Aroclor - 1260												

* SEE EXHIBIT E, PART 7

** CONF. = CONFIRMATION (<20% DIFFERENCE)
 QUANT. = QUANTITATION (<15% DIFFERENCE)

FORM IX

7/85

Pesticide/PCB Identification

Case No. _____ Laboratory _____

Contract No. _____

[illegible]

EXAMPLE INORGANIC DATA REPORTING FORM

(it is likely that parameters will be
added or deleted)

U.S. EPA Contract Laboratory Program
Sample Management Office
P.O. Box 818 - Alexandria, VA 22313
703/557-2490 FTS: 8-557-2490

Date _____

COVER PAGE
INORGANIC ANALYSES DATA PACKAGE

Lab Name _____

Case No. _____

SOW No. _____

Q.C. Report No. _____

Sample Numbers

<u>EPA No.</u>	<u>Lab ID No.</u>	<u>EPA No.</u>	<u>Lab ID No.</u>
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

Comments: _____

Footnotes:

NR - not required by contract at this time

Form I:

Value - If the result is a value greater than or equal to the instrument detection limit but less than the contract required detection limit, report the value in brackets (i.e., [10]). Indicate the analytical method used with P (for ICP/Flame AA) or F (for furnace).

U - Indicates element was analyzed for but not detected. Report with the detection limit value (e.g., 10U).

E - Indicates a value estimated or not reported due to the presence of interference. Explanatory note included on cover page.

s - Indicates value determined by Method of Standard Addition.

R - Indicates spike sample recovery is not within control limits.

* - Indicates duplicate analysis is not within control limits.

+ - Indicates the correlation coefficient for method of standard addition is less than 0.995

Form I

U.S. EPA Contract Laboratory Program
Sample Management Office
P.O. Box 818 - Alexandria, VA 22313
703/557-2490 FTS: 8-557-2490

EPA Sample No. _____

Date _____

INORGANIC ANALYSIS DATA SHEET

LAB NAME _____

CASE NO. _____

SOW NO. _____

LAB SAMPLE ID. NO. _____

QC REPORT NO. _____

Elements Identified and Measured

Concentration: Low _____ Medium _____
Matrix: Water _____ Soil _____ Sludge _____ Other _____

ug/L or mg/kg dry weight (Circle One)

1. <u>Aluminum</u>	13. <u>Magnesium</u>
2. <u>Antimony</u>	14. <u>Manganese</u>
3. <u>Arsenic</u>	15. <u>Mercury</u>
4. <u>Barium</u>	16. <u>Nickel</u>
5. <u>Beryllium</u>	17. <u>Potassium</u>
6. <u>Cadmium</u>	18. <u>Selenium</u>
7. <u>Calcium</u>	19. <u>Silver</u>
8. <u>Chromium</u>	20. <u>Sodium</u>
9. <u>Cobalt</u>	21. <u>Thallium</u>
10. <u>Copper</u>	22. <u>Tin</u>
11. <u>Iron</u>	23. <u>Vanadium</u>
12. <u>Lead</u>	24. <u>Zinc</u>
Cyanide _____	Percent Solids (%) _____

Footnotes: For reporting results to EPA, standard result qualifiers are used as defined on Cover Page. Additional flags or footnotes explaining results are encouraged. Definition of such flags must be explicit and contained on Cover Page, however.

Comments: _____

Lab Manager _____

Form II

Q. C. Report No. _____

INITIAL AND CONTINUING CALIBRATION VERIFICATION³

LAB NAME _____

CASE NO. _____

SOW NO. _____

DATE _____

UNITS _____

Compound		Initial Calib. ¹			Continuing Calibration ²					Method ⁴
Metals:		True Value	Found	ZR	True Value	Found	ZR	Found	ZR	
1.	Aluminum									
2.	Antimony									
3.	Arsenic									
4.	Barium									
5.	Beryllium									
6.	Cadmium									
7.	Calcium									
8.	Chromium									
9.	Cobalt									
10.	Copper									
11.	Iron									
12.	Lead									
13.	Magnesium									
14.	Manganese									
15.	Mercury									
16.	Nickel									
17.	Potassium									
18.	Selenium									
19.	Silver									
20.	Sodium									
21.	Thallium									
22.	Tin									
23.	Vanadium									
24.	Zinc									
Other: _____										
Cyanide										

¹ Initial Calibration Source _____ ² Continuing Calibration Source _____

³ Control Limits: Mercury and Tin 80-120; All Other Compounds 90-110

⁴ Indicate Analytical Method Used: P - ICP/Flame AA; F - Furnace

Form III

Q. C. Report No. _____

BLANKS

LAB NAME _____

CASE NO. _____

DATE _____

UNITS _____

Matrix _____

Preparation Compound	<u>Initial</u>	<u>Continuing Calibration</u>				<u>Preparation Blank</u>	
	<u>Calibration</u>	<u>Blank Value</u>					
	Blank Value	1	2	3	4	1	2
Metals:							
1. <u>Aluminum</u>							
2. <u>Antimony</u>							
3. <u>Arsenic</u>							
4. <u>Barium</u>							
5. <u>Beryllium</u>							
6. <u>Cadmium</u>							
7. <u>Calcium</u>							
8. <u>Chromium</u>							
9. <u>Cobalt</u>							
10. <u>Copper</u>							
11. <u>Iron</u>							
12. <u>Lead</u>							
13. <u>Magnesium</u>							
14. <u>Manganese</u>							
15. <u>Mercury</u>							
16. <u>Nickel</u>							
17. <u>Potassium</u>							
18. <u>Selenium</u>							
19. <u>Silver</u>							
20. <u>Sodium</u>							
21. <u>Thallium</u>							
22. <u>Tin</u>							
23. <u>Vanadium</u>							
24. <u>Zinc</u>							
Other: _____							
Cyanide							

Form V

Q. C. Report No. _____

SPIKE SAMPLE RECOVERY

LAB NAME _____

CASE NO. _____

DATE _____

EPA Sample No. _____

Lab Sample ID No. _____

Units _____

Matrix _____

Compound	Control Limit	Spiked Sample	Sample	Spiked	Z ^R ¹
	Z ^R	Result (SSR)	Result (SR)	Added (SA)	
Metals:					
1. Aluminum	85-115				
2. Antimony	"				
3. Arsenic	"				
4. Barium	"				
5. Beryllium	"				
6. Cadmium	"				
7. Calcium	"				
8. Chromium	"				
9. Cobalt	"				
10. Copper	"				
11. Iron	"				
12. Lead	"				
13. Magnesium	"				
14. Manganese	"				
15. Mercury	80-120				
16. Nickel	85-115				
17. Potassium	"				
18. Selenium	"				
19. Silver	"				
20. Sodium	"				
21. Thallium	"				
22. Tin	"				
23. Vanadium	"				
24. Zinc	"				
Other: _____					
Cyanide	"				

¹ Z^R = [(SSR - SR)/SA] x 100

"R"- out of control

Comments: _____

Form VI

Q. C. Report No. _____

DUPLICATES

LAB NAME _____

CASE NO. _____

DATE _____

EPA Sample No. _____

Lab Sample ID No. _____

Units _____

Matrix _____

Compound	Control Limit ¹	Sample(S)	Duplicate(D)	RPD ²
Metals:				
1. Aluminum				
2. Antimony				
3. Arsenic				
4. Barium				
5. Beryllium				
6. Cadmium				
7. Calcium				
8. Chromium				
9. Cobalt				
10. Copper				
11. Iron				
12. Lead				
13. Magnesium				
14. Manganese				
15. Mercury				
16. Nickel				
17. Potassium				
18. Selenium				
19. Silver				
20. Sodium				
21. Thallium				
22. Tin				
23. Vanadium				
24. Zinc				
Other: _____				
Cyanide				

* Out of Control

¹ To be added at a later date.

$$^2 \text{ RPD} = [|S - D| / ((S + D) / 2)] \times 100$$

NC - Non calculable RPD due to value(s) less than CRDL

Form VII

Q.C. Report No. _____

INSTRUMENT DETECTION LIMITS AND

LABORATORY CONTROL SAMPLE

LAB NAME _____

CASE NO. _____

DATE _____

LCS UNITS ug/L mg/kg

(Circle One)

Compound	Required Detection	Instrument Detection		Lab Control Sample		
	Limits ug/l	Limits (IDL)-ug/l		True	Found	ZR
Metals:		ICP/AA	Furnace			
1. Aluminum						
2. Antimony						
3. Arsenic						
4. Barium						
5. Beryllium						
6. Cadmium						
7. Calcium						
8. Chromium						
9. Cobalt						
10. Copper						
11. Iron						
12. Lead						
13. Magnesium						
14. Manganese						
15. Mercury						
16. Nickel						
17. Potassium						
18. Selenium						
19. Silver						
20. Sodium						
21. Thallium						
22. Tin						
23. Vanadium						
24. Zinc						
Other:						
Cyanide						

Q.C. Report No. _____
STANDARD ADDITION RESULTS

CASE NO. _____

UNITS

[illegible]

+ - correlation coefficient is outside of control window of 0.995.

QC Report No. ...

LAB NAME _____

DATE _____

CASE NO. _____

[illegible]

IFB Amend One.